

**Investigation on Lost Circulation Materials (LCM) by Palm Oil Fronds**

By

**Norazwan Bin Mohamad Yusak**

**10288**

Dissertation submitted in partial fulfillment of  
the requirements for the  
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(Petroleum Engineering)

JAN 2011

Universiti Teknologi PETRONAS  
Bandar Seri Iskandar  
31750 Tronoh  
Perak Darul Ridzuan

**CERTIFICATION OF APPROVAL**

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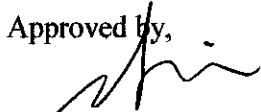
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Petroleum Engineering Programme  
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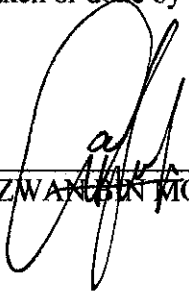


**(Mr Elias B. Abillah)**

**UNIVERSITI TEKNOLOGI PETRONAS  
TRONOH, PERAK  
JAN 2011**

## **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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NORAZWAN BIN MOHAMAD YUSAK

## **ABSTRACT**

This report discuss about the method of the invention and several studies about lost circulation material. The objective on this project is to proposed palm oil fronds as drilling fluid additive to control the lost circulation during the drilling program. Malaysia is the second largest of exporter palm oil in the world and by this invention the abandon of palm oil fronds can be used as lost circulation material. The report discusses of the lost circulation problems, the lost circulation materials and the characterization of the parameters of drilling fluid. It also confers the methodologies for LCM to resolve severe lost circulation problems and designing the mud samples, the mud densities, the rheological tests, and the filtration performance volume tests. Tests have been conducted in accordance with the API 13 B and the related equipments were mixer, mud balance and viscometer. Properties measured through this experiment such as density, plastic viscosity, yield point, 10 seconds and 10 minutes gel strength and filtration rate. The experiment shows that, palm oil fronds have characteristic as lost circulation material as comparing with nut plug and corn cob. This report present the data collection, analysis and discussion of results obtain.

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## **CHAPTER 1: INTRODUCTION**

### **1.1 Background of Study**

The key to making the rotary drilling system work is the ability to circulate a fluid continuously down through the drill pipe, out through the bit nozzles and back to the surface. The drilling fluid can be air, foam which a combination of air and liquid or a liquid. Liquid drilling fluids are commonly called drilling mud. All drilling fluids, especially drilling mud, can have a wide range of chemical and physical properties. These properties are specifically designed for drilling conditions and the special problems that must be handled in drilling a well.[1]

Drilling fluid are mixture of natural and synthetic chemical compounds used to cool and lubricate the drill bit , clean the bottom hole, carry cutting to the surface, control formation pressure, and improve the function of the drill string and tool in the hole. There have 2 type of drilling fluid, water based mud and oil based mud. The type of fluid based on drilling operation needed. In oil well drilling, various problems related to drilling fluid properties arise. Some of these problems such as slow drilling rates or excessive drilling pipe torque. For this study more on lost circulation problems. Lost circulation problem can interrupting the drilling progress for weeks and sometimes lead to abandonment of well.[2]

Lost circulation materials are use to prevent mud losses to the wellbore. The consequences of lost circulation can be as little as the loss of a few dollars of drilling fluid or as disastrous as a blowout and loss of life, so close monitoring of tanks, pits, and flow from the well, to quickly assess and control lost circulation, is taught and practiced. If the fluid in the wellbore drops due to lost circulation ,hydrostatic pressure is reduced, thus allowing a gas or fluid, which is under a higher pressure than the reduced hydrostatic pressure, to influx into the wellbore.

Another significance of lost circulation is called dry drilling. Dry drilling occurs when fluid is completely lost from the well bore without actual drilling coming to a stop. The effects of dry drilling can be as slight as destroying a bit as serious as major problem to the wellbore requiring a new well to be drilled. Dry drilling can also cause severe damage to the drill string, including snapping the pipe, and the drilling rig itself.

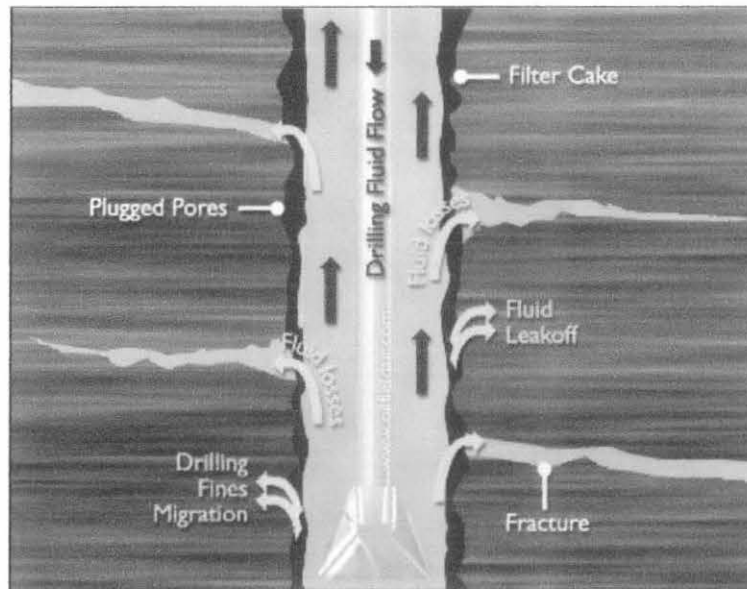
These problems can be counter by added or put the lost circulation material to prevent the drilling fluid enter the formation. The chemical or material will act as cement in the wall wellbore. So, the mud can circulate to the mud pit at same amount before continue drilling.

## **1.2 Problem Statement**

Lost circulation is common problem in drilling a wellbore. Lost circulation is a subterranean loss of drilling fluid while drilling. Certain type of geological formation are porous and during drilling the drilling fluid have tendency to migrate into formation rather than travelling into the annulus between the drill string and the formation wall and eventually emerging at the surface. These losses of drilling fluid become costly. In traditional method in oil and gas industry, they use several chemical to prevent mud losses to the formation. There have two categories of losses in field such as:

- "Minor losses" - This is where the losses are between 6 and 470 barrels, and remains within those amounts, or is ceased, within 48 hours.
- "Severe losses" - This is where the losses are greater than 470 barrels , or it takes greater than 48 hours to control or cease the lost circulation.

Total losses also occur in the drilling program, where the return of fluids is completely lost to the surface. This may fall into either the minor or severe losses categories, depending on the amount of losses and the time involved in regaining circulation.



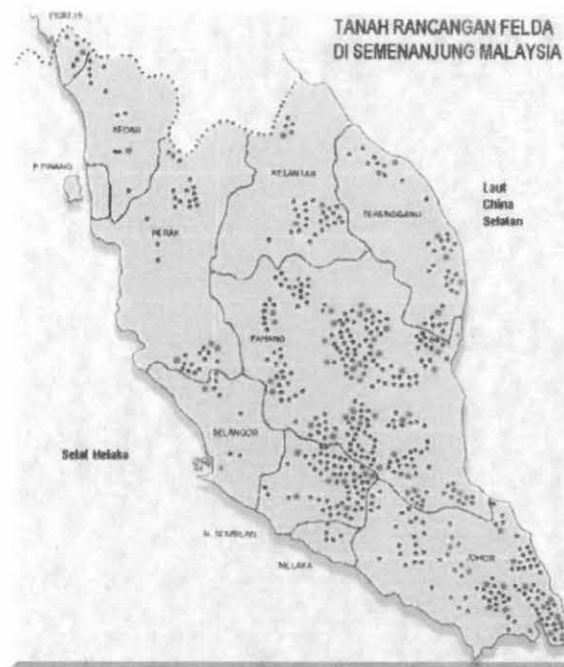
**Figure 1: Lost circulation problem.**

Normally loss zones are classified in drilling industry as seepage loss about 110bbl/hour, partially loss about 10-500bbl and completely lost around 500bbl/hr(Suyan,2007).Consequent upon industry about this problem, they has developed the following basic type of agents depending on the operational phase of the field to combat the loss problem.

- Bridging agent
- Gelling agents
- Cementing agents

Generally three type of lost circulation agent have been frequently used in industry to combat lost circulation while drilling. The bridging agents are found effective to combat loss circulation problem and they classified in term of morphology.

Some of the LCM products are not compatible with drilling fluid that we developed. It may sag in the mud after a few minute. We can identify the sagging during the rheology test on gel strength. If mud that we are formulated is sag, it may cause lost efficiency of the drilling fluid. It can due to the rig problem such as, bit balling, stuck pipe and formation damage because cutting that we drill cannot transfer to the surface. These mean our objective to bring the cutting to the surface are fail.



**Figure 2: Distribution of palm oil tree in peninsular Malaysia**

### 1.3 Objective and Scope of Study

Objective of this project is to develop a new lost circulation material which more compatible with drilling fluid and the environmental friendly. Even though there have several lost circulation material that was available in the market, but it is not compatible and difficult to get a big amount of quantity. In other hand, the price for the lost circulation material is quite expensive. Those because several lost circulation material are limited in term of the usage. For instance like starch, even though it is environmental friendly additive but it still limited resource because most of the starch we use to make bread and for daily uses.

So, in this project we try to use the palm oil fronds as the lost circulation material in the oil field. Why we choose palm oil fronds? That because palm oil fronds are easy to get from the settler or farmer and at the same time we can get with a big amount. The mature trees are single-stemmed, and grow to 20 m tall. A young tree produces about 30 fronds a year. Conventionally for trees over 10 years produce about 20 leaves a year. The leaves are pinnate, and reach between 3-5 m long. It is available in any palm oil farm in this country. At the same time if we can produce the lost circulation material by using palm oil fronds, it can increase the socioeconomic for the farmer. Normally when the farmer harvest the frond they will burn the fronds

and it will effect to our environment in term of air pollution. So that in order we can increase the economics of the farmer we also can save our world form the pollution disaster.



**Figure 3: Palm oil tree.**

In order to achieve the objective of this project, several parameter that we need to study and do comparison between current drilling fluids. The parameter such as:

- Plastic viscosity
- Yield point
- Gel strength
- API filter press(filtration)
- Plugging Permeability Test (PPT)
- Thickness of filter cake
- pH of drilling fluid
- Density of drilling fluid

Based on this parameter, we use to compare the drilling fluid by using palm oil fronds as loss circulation material and common drilling fluid formulation. So that, we can evaluate the performance and effect of the palm oil fronds in drilling fluid.

## **1.4 Tools required for the project.**

For this study several item that we use to run the test for the project. The tools or testing equipment that we are going to use such as:

### **1.4.1 Viscometer (Fann 35)**

Viscometer is use to measure the viscosity of the fluid. For this project we proposed to use the Fann 35 viscometer. This instrument with ability to test at six different speeds rotation. The range of the speed is from 3 rpm up to 600rpm with the speed determined by a combination of speed switch setting and viscometer gear knob placement. To select the desired speed, set the speed switch located on the right side on the base to high or low speed position desired. Then turn the motor on and move the viscometer gear shift knob placed in the centre of the top of the instrument to desired position. From the reading of the fann viscometer we can measure plastic viscosity, yield point and gel strength

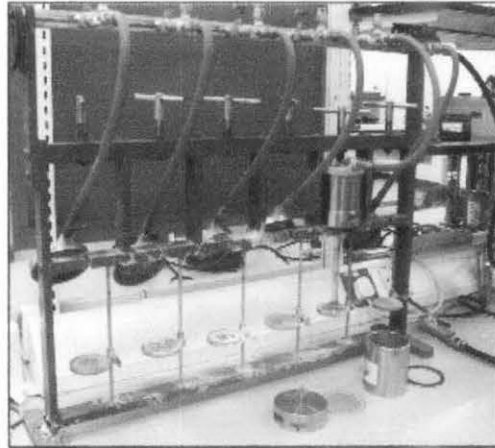


**Figure 4: Viscometer (Fann 35)**

### **1.4.2 API filtration**

API filter press is equipment that we use to measure the filtration of our drilling fluid. There are many different methods of filtration. All aim to attain the separation of substances. Separation is achieved by some form of relations between the substance or objects to be removed and the filter. The substance that is to pass throughout the filter must be a fluid. Methods of filtration vary depending on the position of the targeted material whether it is dissolved in the fluid phase or

suspended as a solid. Filtration also the passage of filtrate in to the formation dies to difference pressure. When we apply the pressure from the top, the solid or particles will block the pore and only a few filtrate or liquid can flow through the paper or formation. In the oilfield, if the solid in the mud cannot plug the pore in the formation, then mud will go through to the formation.



**Figure 5: API filtration press**

#### **1.4.3 Aging cell**

Aging cell is an equipment that to simulate drilling fluid in well condition. We put the drilling inside the aging cell and pressurised with certain pressure. Then we roll the cell in the rolling oven.

#### **1.4.4 Rolling oven.**

Any fluid used in a well for the purpose of carrying either lifting or transporting, aggregate materials, i.e. drilling muds, lost circulation fluids, fracturing fluids, etc, should be evaluated for stability at temperature with time. Also it is important to determine the devicosifying T (break time) for any polymer fluids which the end-user does not want to remain in the well.

Permanently to decide when is the best time to open the well and flow the broken gelled fluid back. Static measurements used for these studies are often not accurate - the roller oven keeps the fluid under constant dynamic conditions for these measurements or observations to be conducted.



#### **1.4.5 Hamilton Beach mixer**

This type of mixer is used to mix quantities up to 1 gal. It has 3 types of speed based upon the mixing procedure. The order of addition and the mixing speed is left up to the discretion of the lab analyst. The mixing order and time is recorded in the event that a reproduction of the result is required.



**Figure 6: Hamilton Beach Mixer**

#### **1.4.6 Pycnometer**

This equipment is used to determine the specific gravity of solid or powder sample by measuring the pressure change of helium within calibrated volumes. This equipment consists of two main parts which are calibration ball and cup and micrometrics automated pycnometer.



**Figure 7: Pycnometer**

#### **1.4.7 Permeability Plugging Test (PPT).**

The Permeability Plugging Tester (PPT) is a adaptation of the standard 500-mL HTHP filter press. It may be used in the field or in a laboratory environment. The instrument is useful for performing filtration tests on plugging materials without the interference of particles settling on the filter medium during the heat up process. The PPT is very helpful in predicting how a drilling fluid can form a low permeable filter cake to seal off depleted, under pressured intervals and help prevent differential sticking. Typical differential pressures are much higher than those seen in standard HTHP testing. The pressure cell is alike to those seen in standard HTHP filtration testing, but it is inverted with the filter medium and the back pressure receiver on top of the assembly.



**Figure8: Permeability plugging test equipment**

## **CHAPTER 2: LITERATURE REVIEW**

### **2.1 Method for reducing lost circulation.**

Another swellable material sometimes used is water expandable clay such as bentonite which may be mixed with another ingredient to form viscous paste cement. Lost circulation made by forming slurry of bentonite and cement oil. The slurry is mixed with a surfactant and water to form composition comprising a water oil emulsion having the bentonite and cement dispersed in the continuous oil phase, this composition is pump down the wellbore, the oil expands and flocculate the bentonite under right condition.[2].

Most method using for control lost circulation material have their disadvantages and making necessary to do more research for effective, efficient and readily controlled method of reducing the loses. When some of the materials mentioned above above do the reach lost circulation zone they must activated to absorb water and swell still in the proper region against difficult to control. According to the current invention they use polymers as additives. The selected polymers are in granular from which can be introduce in wellbore along water based mud drilling fluid. One in the lost circulation area, the granules will accumulates absorb the water and swell to form 'soft gel' and sealing porous formation.

### **2.2 Test for lost circulation material.**

Test has been devised to determine the usefulness of a particular material as control additives with respect of the two type of lost circulation causing formation. The 'slot test' is standard practise used to determine control practise with respect to the formation. The purpose of this test is to observe the ability of a drilling fluid which under pressure containing the control candidates to plug a slit of predetermined dimension. The 'shot bed test' observes the ability of the drilling fluid to plug the microporous formation by having mud containing the control candidates to attempt to pass through column of shot of standard size.

Current lost circulation material controller are generally divide into three different categories which are fibers, flake and granular. Each category have their own specific function with respect of formation. Material which are in the form of fibers and flakes presently used to seal microporous formation. Example of fibrous material commonly used includes cedar fibers and bagasse. Example of flakes materials

commonly used includes paper, mica and cottonseed hulls. Granular materials such as ground walnut or pecan shells are common used to plug the fracture formation. Therefore the circulation control using presently known materials requires the knowledge of the nature of the subterranean formation or use of type of combination of material until mud circulation is substantially restored.

### 2.2.1 Core injection tests.

For this test in journal, they use to evaluate the lost circulation material between water based mud and oil based mud system. This simulation is done by using natural or sythetic core of 1 inch diameter by 6 inch long. The composition of core were limestone and sandstones with the permeability varying 0.1 to 6 Darcy. In order to evaluate the any compatible problem with water or oil based fluid, the core were saturated with the fresh water, brine and diesel. Pumping is simulated by stirring the mixture at the test temperature in an autoclave under pressure for 60%to 90% of the design isothermal gel time before pumping the core at constant differential pressure. Before pumping the mixture a preflush equivalent to one pore volume first injected which is fresh water for the brine saturated cores and fresh water plus 2.5% by volume surfactant to the diesel saturated cores. Pumping through the core stop due to the increase of the mixture viscosity. Then, left the system for one hour under static condition before attempting to restore flow through the core by increasing the differential pressure.

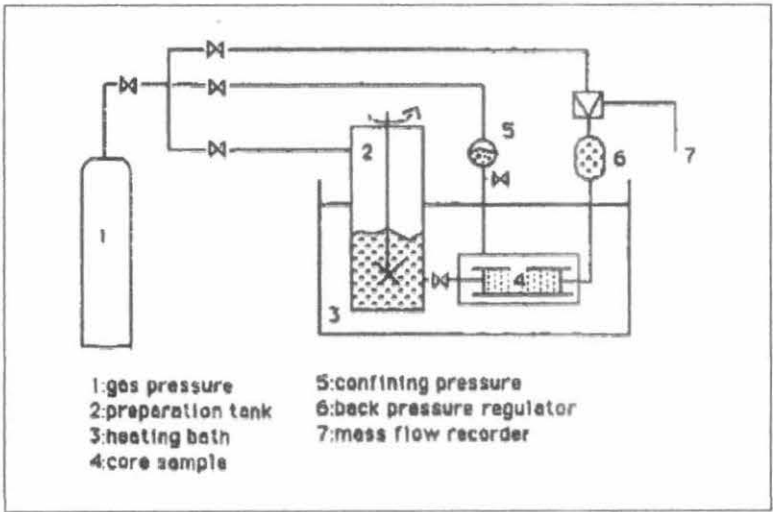


Figure 9: Experiment setup for core injection flow.

### **2.3 Example lost circulation using cocoa bean shells.**

A based mud was prepared using a commercially available viscosifier sodium bentonite.[2] The bentonite was added to water under high speed stirring. The mud was allowed to stand overnight and then adjusted to a viscosity of 95cp. Cocoa bean shells were ground using a 24-inch single runner attrition mill. The materials were passed through a series of U.S. standard series sieves of the fine series and the separate portions were recombined to give a mixture having a particle size range of from 4 to 100 mesh in the following proportions. The sized and ground cocoa bean shell materials were microscopically observed to be irregular shape with a surface having a number of 'hills', 'knobs' and 'ridges' and that particles were generally of a configuration in which one dimension was substantially smaller than other.

The shot bed test cell consisted of a tail filter cell in which the bottom had a 3/16 inch ID outlet. A 14 mesh screen was placed on the bottom of the cell and covered with a 0.75 inch bed of N0.8 lead shot. This cell simulated sealing characteristics in a macroporous formation. The slot test cell was formed from a tail filter cell in which the bottom contained a slot of 2 inch long, 0.25 inch deep and adjusted to a width of 0.05 inch. This cell simulated sealing characteristics with respect to fracture formation.

Separate 500ml samples containing varying amounts of the particulate cocoa bean shell mixtures described above were each tested in a lost circulation control cell, applying 100psi pressure of N<sub>2</sub> and observed the time to seal the cell and the volume of mud collected prior to seal.

### **2.4 Carbon particle in lost circulation materials.**

Carbon particles are often added to drilling fluids for the purpose of reducing the frictional resistance of the drill pipe in contact with the borehole wall. The carbon particles embed on the cake or mat formed at the wall of the borehole over permeable formation drilled during operation. It is believed that the carbon particles serve as a lubricant in the cake. This lower frictional resistance is very useful in preventing sticking of the pipe in the wall during the drilling operation. Especially on the high permeable formations in contact with the borehole. Therefore the carbon particles enhance the benefit of the fluid loss additives.

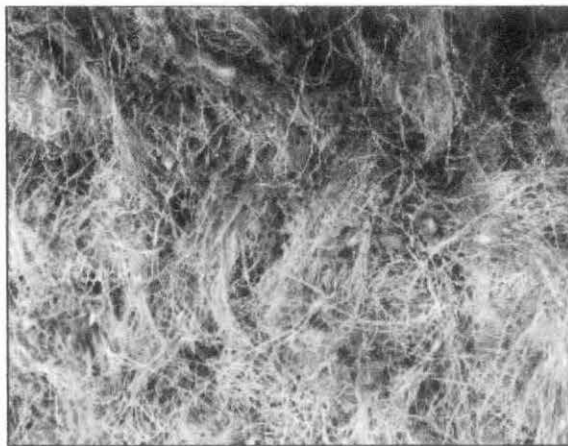
## **2.5 Size and type of lost circulation material.**

A wide assortment of bridging agent or plugging material is available for reducing lost circulation or restoring circulation during cementing a well. For the purpose of classification, the lost circulation material can divide in 3 group classification; fibers, flakes, granules.

### **2.5.1 Fibrous LCM's**

When add the drilling mud with the fibrous LCM, it will form mat-like bridge over porous formation (Nayberg, 1986). This mat effect the reduction of the opening to the formation, permitting the colloidal particles in the mud to rapid deposit the filter cake, complexly sealing the formation that otherwise make require the cementing job.

The maximum size of the gradation of the size is more important to their performance that composition. The physical and chemical nature of the material however has certain limitation as to the size gradation obtainable, resistance to disintegration, and degradation when circulated in the mud system. The maximum size of leather and asbestos fibers for example is considerably smaller that can be obtained with bagasse or bark fibers.

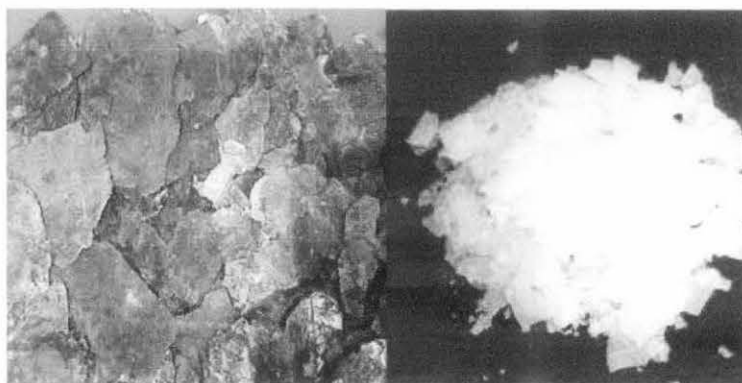


**Figure 10: Fibrous lost circulation materials**

### **2.5.2 Flake LCM's**

The example of flake type LCM includes mica, cork, corn cob and cellophane. Cellophane flake are composed of 3/8" to 3/4" in graded cellulose or polyvinyl film flakes. The products that common used in field are cellophane and

mica in coarse and fine grade. The flake types LCM are design to bridge and form mat on the formation face. This material can plug and bridging many types of porous formation to stop the mud losses.



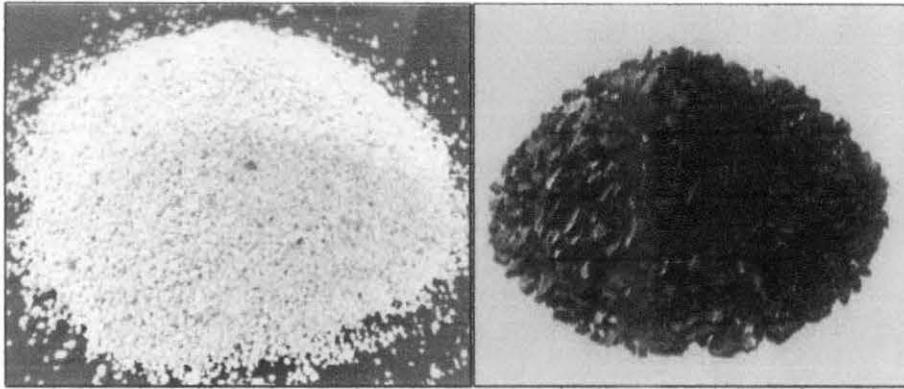
**Figure 11: Flakes lost circulation materials**

### **2.5.3 Granular LCM's**

Granular LCM include the walnut shells, gilsonite, perlite wood coke and asphalt. The granular LCM's form two type of bridges:

- At the formation face
- Within formation matrix.

The latter type of sealing is preferred a more permanent bridge forms within the formation and the LCM's do not become dislodge easily as the result of pipe movement in the wellbore. (Nayberg, 1986).The effectiveness of granular is depend on proper particle size distribution to build the bridge having decreasing the permeability. A blend of large, medium, small particles or a blend of relatively large and small particle are commonly use. It is believed that the particle will transport to the formation during the bridging occur. The granular LCM normally work better in high solid ration system such as cement slurry compare in the drilling mud system.



**Figure 12: Granular Lost circulation materials**

#### **2.5.4. Blended LCM's**

Blended LCM is blend of two or more lost circulation material in the drilling fluid. It has proven the effectiveness in the field. A popular blended LCM's furnishes the advantage of having the gradation of particle size and variation of type of material blended in a one sack mixture to provide effective sealing. This blending LCM's is a combination of granular, flakes, fibrous material with varying the size and shape. This blend will penetrate the fracture, vugs or extremely permeable zones and seal them off effectively.

### **2.6 Effect of Material Type and Size Distribution on Performance of Loss Control Material**

On this literature discuss about evaluate and compare the performance af the different LCM and determine the effect of size distribution on their performance. Twenty four different LCM made from various materials and blends of material having different size distribution were tested with three different based mud[23]. The author use permeability plugging test(PPT) to evaluate the filtration of LCM. From the experiment it show the effect and performance of LCM based on the mud type, LCM materials, concentration and size distribution. This paper also tells us about the procedure of testing loss circulation materials and discussion about the effects.

#### **2.6.1 Materials**

Based on the paper, twenty four different sample were evaluated with different type of drilling fluid. The sample use in this studies include fine, granular and fibrous LCM. All the sample will mix together with the three different drilling



fluid and observe the performance for each type of drilling fluid. All the drilling fluid were weighted to density about 14-15 ppg. The samples were use given in Table 1.

Sample	Contents	Sample	Contents
1	Corncob fine	13	Kenaf fine/wood floor
2	Corncob medium	14	kenaf fine/almond shell fine
3	almond shgell fine	15	kenaf fine/inner shell fine
4	Inner shell fine	16	corncob coarse
5	Inner shell medium	17	Almond shell coarse
6	Wood floor	18	Kenaf coarse
7	corncob/ inner shell fine	19	Proprietary seepage circulation materials
8	wood floor/inner shell fine	20	Proprietary lost circulation materials
9	wood floor/inner shell fine	21	Cedar fiber fine
10	Kenaf fine/ wood floor	22	Proprietary seepage control material #2
11	woodfloor/inner shell fine/inner shell medium	23	100 hard woody ring
12	Kenaf fine	24	proprietary seepage control material #3

Table 1: twenty-four sample use in lost circulation test

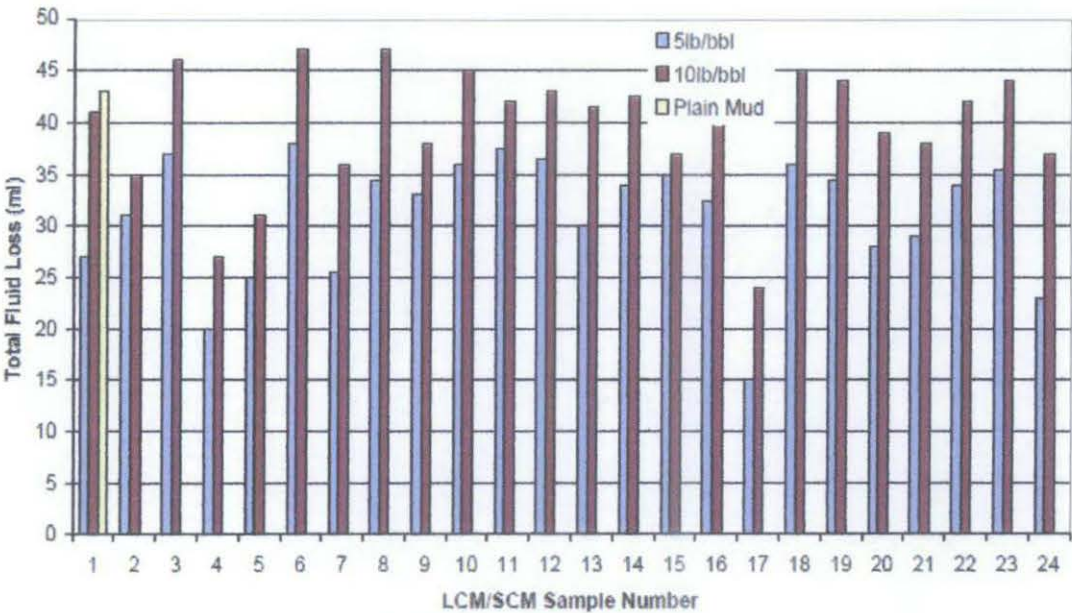
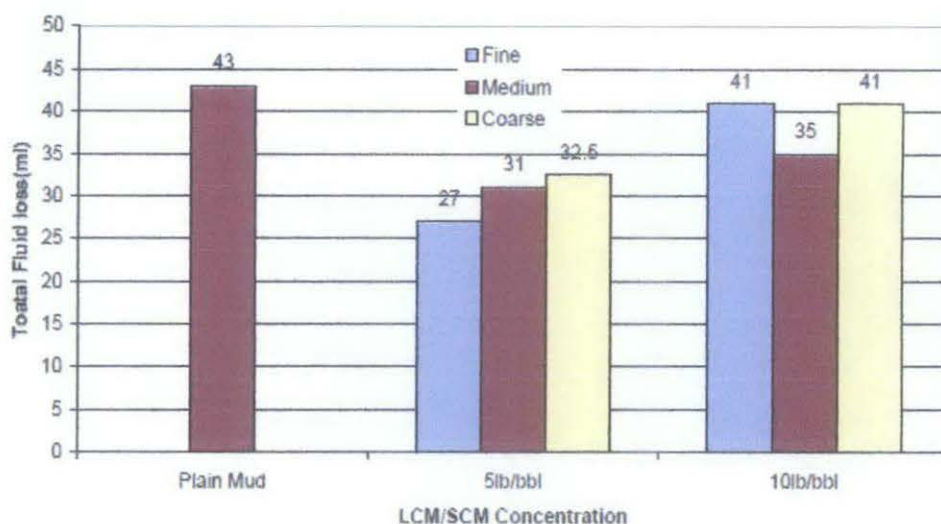


Figure 13: Total Fluid loss for 24 sample materials.



**Figure 14: Effect of LCM on total fluid loss for corn cob sample**

## 2.6 Palm oil Frond composition.

A palm branch or palm fronds usually refer to the leaves of the *Areaceae*. The palm oil fronds mainly composed of cellulose, hemicelluloses and lignin. In addition to those three major components, some percent of ash and extractives are included. Among those components, cellulose and hemicelluloses are polysaccharides, which are simply decomposed and metabolized by wood-rot fungi. In the constituent analysis of wood, when lignin is selectively removed, what obtain is holocellulose. Holocellulose can be consideration of as the total of cellulose and all hemicelluloses (KOZUMAYosei *et al.*).

Fiber Property of Oil Palm Fronds	
Mean fiber length (mm)	
Arithmetic	0.59
Length weighted	1.13
Weight weighted	1.54
Coariness (mg/mm)	0.098
Fiber dimensions (um)	
Fiber diameter	19.6
Lumen length	11.6
Cell wall thickness (T)	3.97

\*source: Law and Jiang (2001)

**Table 2: Fiber characteristic of palm oil fronds**

Lignin of oil palm frond was characterized by the existence of significant amounts of esterified *p*-hydroxybenzoic acid together with small amounts of etherified *p*-hydroxybenzoic acid. Vanillic and siring acids were esterified or etherified to lignin. Some extents of these ester bonds and *beta*-O-4 inter unit linkages of lignin were cleaved during steam explosion, in addition to great condensation of *guaiacyl* nuclei, as revealed by <sup>1</sup>H- and <sup>13</sup>C-NMR spectra of isolated lignin from the steam exploded pulps, of which yields were quite high, suggesting that lignin has been released from other wall polymers. Wall polysaccharides of oil palm frond are composed of cellulose and considerably high concentration of *arabinoxylan*, which produced great abundance 5-hydroxymethyl-furfural and furfural during steam explosion, respectively and even hot pressing at 125°C to prepare binderless boards. It is suggested that released lignin and furfural derivatives generated during steam explosion give to self-binding of the steam exploded pulps. However, severe situation of steam explosion caused great damages in lignin macromolecules, and gave poor feature of binderless board.

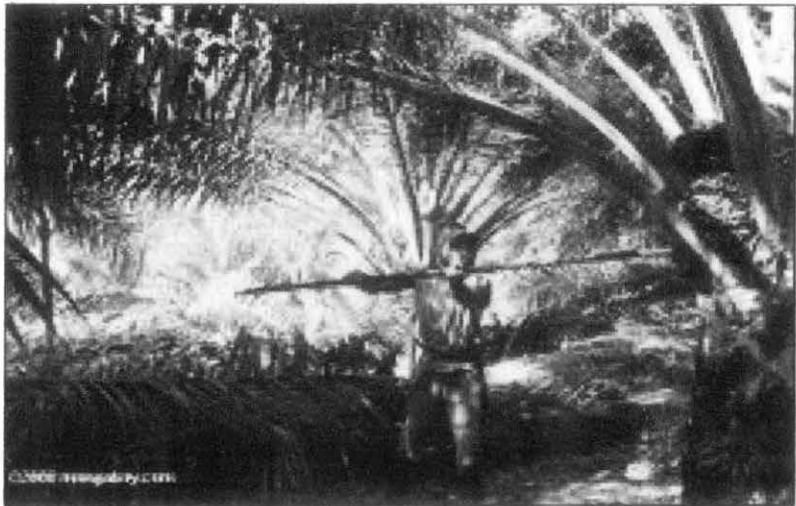
Component	composition
Lignin	15.2
Holocellulose	82.2
Alpha cellulose	47.6
Ash	0.7
<b>Polysaccharide Composition</b>	
Arabinose	1.5
Mannose	2.2
Galactose	0.9
Glucose	66.6
Xylose	28.9

**Table 3: Chemical composition of palm oil Fronds**

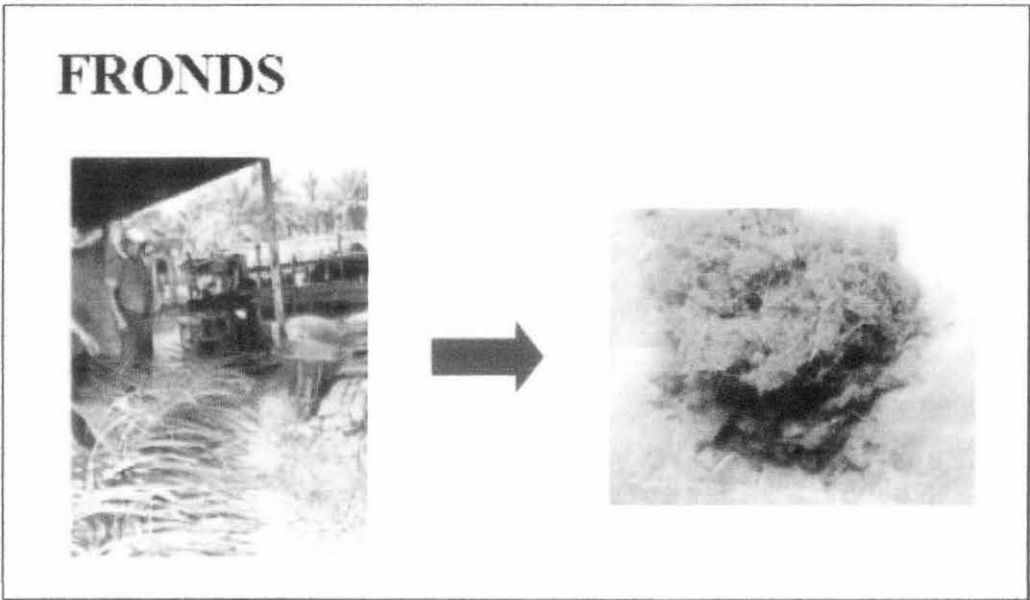
### 2.6.1 Harvesting of the fronds

It is an agricultural plant, which originate starting from West Africa and cultivated in Malaysia for its oil producing fruit. Besides palm oil, the industry also generates huge amounts of lignocelluloses residues such as trunks, fronds and the empty fruit bunches, with an estimated amount of 30 million tonnes (MPOB, 2001). The suitability of this abundant, low-priced and renewable unprocessed material for

papermaking resource has been explored using a variety of pulping methods. Mostly, fronds are taken from palm oil waste which is usually will be burn at the farm.



**Figure 15: Harvesting the palm oil fronds.**



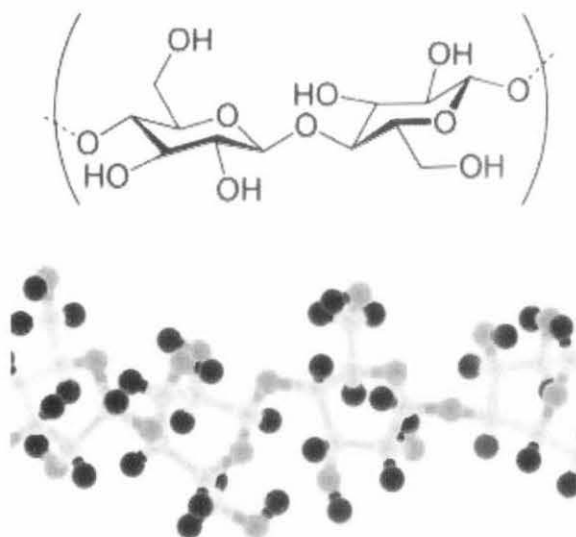
**Figure 16: Frond process to be the fibrous LCM**

**2.7 Cellulose**

Cellulose is an organic composite with the formula  $(C_6H_{10}O_5)_n$ . It is a structural polysaccharide resulting from beta-glucose. Cellulose is the main structural component of green plants. The main cell wall of green plants is made of cellulose; acetic acid bacteria are also known to synthesize cellulose, as well as many forms of algae, and the oomycetes. Cellulose was exposed and isolated in the mid-nineteenth

century by the French chemist Anselme Payen with an estimated annual production of  $1.5 \times 10^9$  Tonnes.

Some animals, particularly ruminants and termites, can digest cellulose with the help of symbiotic micro-organisms - see methanogen. Cellulose is not eatable by humans and is often referred to as 'dietary fiber' or 'roughage', acting as a hydrophilic bulking agent for feces.



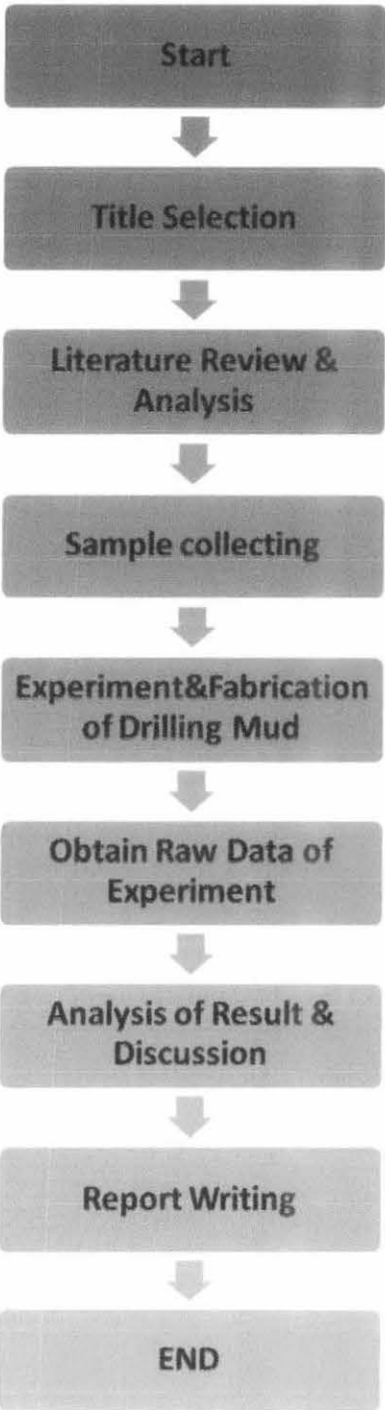
**Figure 17: Molecule Structure of Cellulose**

Cellulose is derived from ( $\beta$ -glucose), which condense through  $\beta(1 \rightarrow 4)$ -glycosidic bonds. This linkage motif contrasts with that for  $\alpha(1 \rightarrow 4)$ -glycosidic bonds present in starch and other carbohydrates. Cellulose is a straight chain polymer: dissimilar with starch, no coiling occurs, and the molecule adopts an extended rod like conformation. In micro fibrils, the multiple hydroxyl groups on the glucose residues hydrogen bond with each other, holding the chains firmly together. In field cellulose is use as the fluid loss controller. So if we refer in the palm oil frond, there contain the cellulose which mean it can help to control the mud filtrate in the field.



**CHAPTER 3: METHODOLOGY**

**3.1 Research Methodology**



**Figure 18: Loss circulation material project methodology**

In order to achieve the aim of the project the methodology of the project consists of the following steps:

- For this study we need to do a survey on current lost circulation material that they use in drilling fluid.
- Find several materials about the function and raw material of loss circulation materials.
- Do research on the properties of palm oil fronds which can be use as loss circulation material. The structure and particle of the palm oil leaves component.
- Make raw material for the loss circulation material by using palm oil fronds. Make different type of shape and size.
- Mix 10.5ppg water based mud with palm oil fronds lost circulation material and mix other drilling fluid with current lost circulation material such as nut plug.
- Test the properties of the drilling fluid by using viscometer and compare the properties of the two drilling fluid.
- Test the API filtration test to collect the filtration.
- Test the permeability plugging test (ppt) as recommended for lost circulation test.

Finally when we got all the data from the experiment we start to do the report.

3.2 Mud design

Mud will design with same amount of lost circulation material and see the result of the test. The picture below is the spread sheet to calculate the additive uses for 10.5ppg mud weight. For this calculation we use water based mud spread sheet.

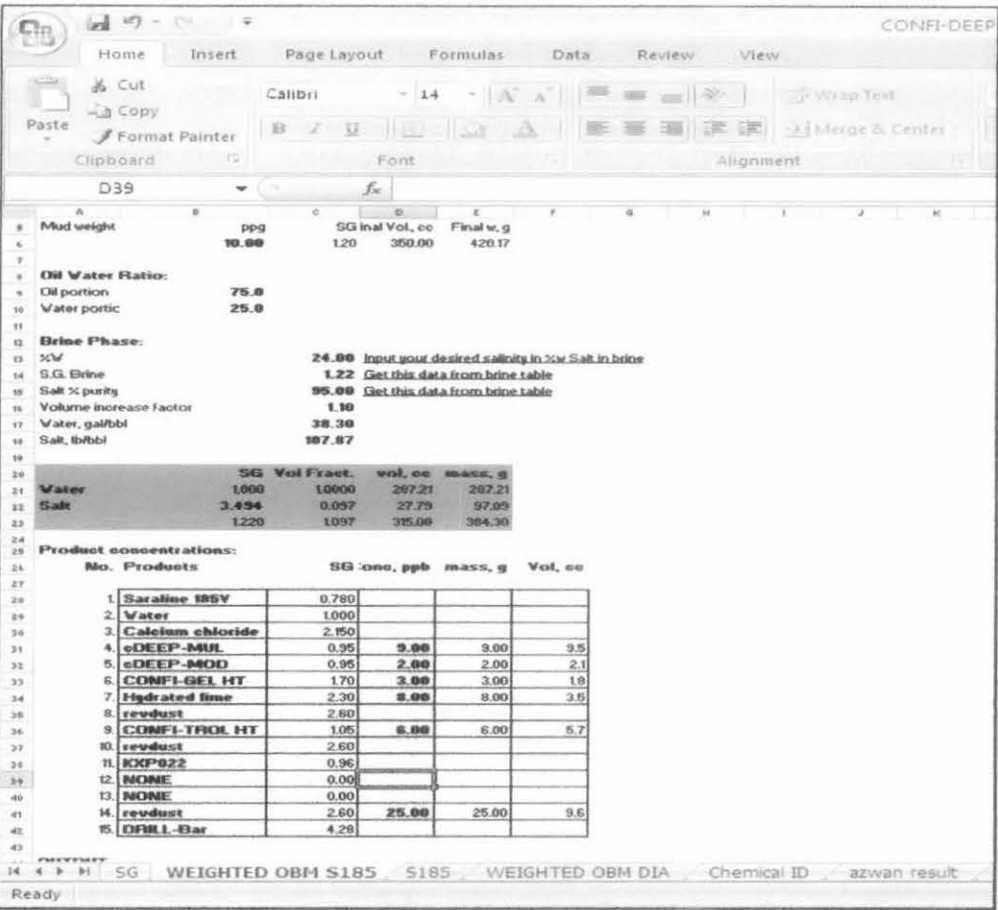


Figure 19: Spread sheet for additive calculation.

Product	Function
Fresh water	As the water based mud system.
Bentonite	Viscosifier
Soda ash	To reduce hardness
Caustic soda	To control the pH
Palm oil fronds	Lost circulation material

Table 4: Mud formulation for LCM test



### 3.3 Mixing Guidelines

		Mixer:	Hamilton	Silverson	
		Mixing Order	Speed	Speed	Mixing time
No.	Product			rpm	min
1	Make up water	1	High	6500	-
2	Soda ash	2	High	6500	2
3	Salt	3	High	6500	2
4	Bentonite	4	High	6500	2
5	Fluid loss additives	5	High	6500	2
6	Viscosifier	6	High	6500	5
7	Shale inhibitor	7	High	6500	2
8	Weighting agent	8	High	6500	2
9	pH control	9	High	6500	2
10	Solid additives	10	High	6500	2

**Table 5: Guideline for mixing water based mud**

### 3.4 Checking mud weight

Determining Mud Weight Using the Mud Balance:

- Instrument base must be set on a flat level surface.
- Measure and record the mud temperature.
- Fill the mud cup with the mud to be tested.
- Replace cap and rotate until it is firmly seated, ensuring some of the mud is expelled through the hole on top, to free any trapped gas.
- Place the beam on the base support and balance it by using the rider along the graduated scale. Balance is achieved when the bubble is directly under the centre line.
- Take the mud weight reading

**The hydrostatic head produced by the mud in psi is =  $0.052 \times G \times H$**

**Where       $G$  = density of mud in ppg**

**$H$  = depth of the hole in feet.**

This hydrostatic head will counter the formation pressure in order to avoid a blowout while drilling.

**3.5 Rheology Test:**

In mud rheology testing, rheometer is used. It is important to frequently monitor the mud rheology as to make sure that the mud is always within the specification as stated in the mud program:

- Place the sample in the rheometer thermo cup and adjust the cup until the mud surface level is equal height to the scribed line on the rotor surface.
- Set the thermometer cup at 120°F and wait until the temperature reaches the set temperature. 120°F is API standard for rheology test of drilling fluid.
- Turn on the rheometer, first taking dial measurements at the top most speed (600rpm), then gradually switch to lower gear and to obtain all readings (600,300,200,100,6,3rpms).

**Determining PV**

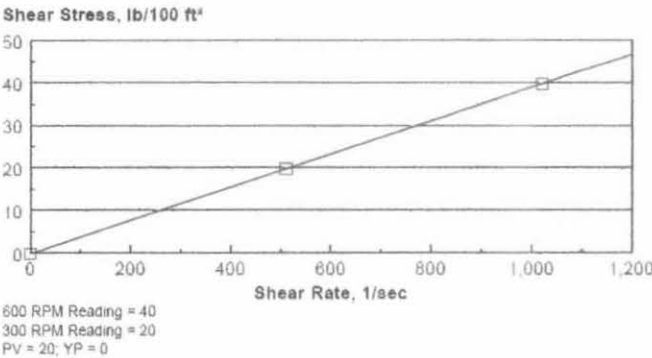
Indicate the amount of solids (sands, silts) in mud. High PV means that the mud is not clean and there is a problem with the solids control equipment.

PV = 600 rpm -300 rpm

**Determining YP**

Indicate the carrying capacity of drilling fluid.

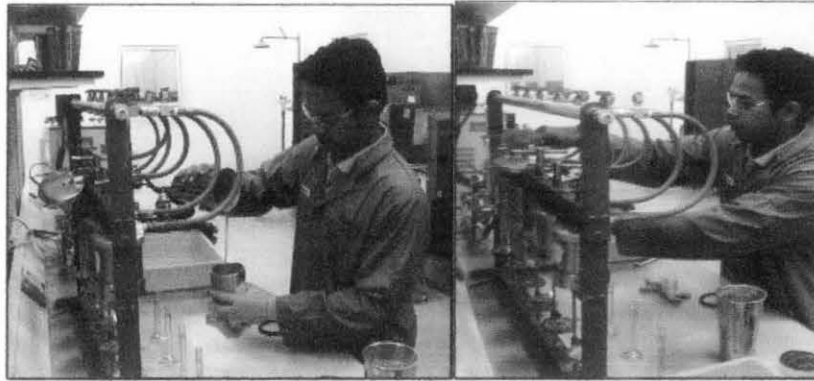
YP = 300 rpm – PV



**Figure 20: Newtonian fluid model**

### 3.6 API filtrate using filter press:

The API filtrate is a test designated to determine the milliliters of filtrate lost in 30 minutes under a 100 psi pressure. Knowing the fluid loss is important since it is bad to have mud that has a high filtrate loss because this contribute to high fluid invasion and also thick mud cakes.



**Figure 21: During API filtration press testing.**

### 3.7 Plugging permeability test (PPT)

After drilling fluid done with API filter press, we run the next filtration test which is plugging permeability test. The filter media employed in PPT is a ceramic disc which can have different permeability. For this test we use 1000 micron of ceramic disc as simulate the formation permeability. It has been suggested that PPT provide better representation of downhole static condition [24]. Basically, PPT is a HPHT fluid loss that has been modified to better simulate well conditions. This is accomplished by positioning the filtration medium above the sample drilling fluid. Pressure is applied from the bottom of the cell by using nitrogen gas and filtrate will collect from the top of the PPT cell. Once the test is running, the first spurt loss was collected and note, and it was followed by collection of filtrate for thirty minutes. The total fluid loss was then calculated according to the recommended procedure by API.

$$\text{Total fluid loss} = \text{spurt loss (ml)} + 2 \times (30 \text{ min. fluid recovery})$$

### **3.8 Gantt Chart**

A Gantt chart is a type of bar chart that illustrates a project schedule. Gantt charts illustrate the start and finish dates of the terminal elements and summary elements of a project. Terminal elements and summary elements comprise the work breakdown structure of the project. Gantt charts are important for a project to make sure the task or the fork structure feasibility with the time frame. Each stage has the specific time frame and we need to follow the time line to make sure the end of the due date, we can produce or done our project. For this project the Gantt chart was attached in appendixes.

**(Appendix 1)**

## CHAPTER 4: RESULT AND DISCUSSION.

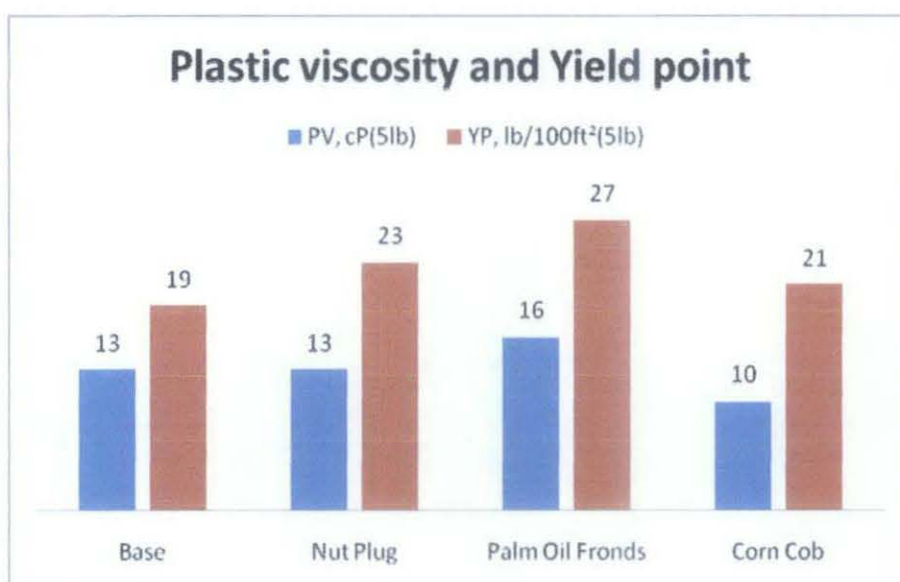
### 4.1 Rheological Properties.

Rheological properties are basic mud characteristic. From the rheological properties we can get the viscosity, yield point and gel strength for our drilling fluid. These properties are important to know our drilling fluid efficiency. Without knowing our rheological properties of our drilling fluid, it can cause a lot problem to our well bore mostly in cutting carrying to the surface. On this experiment, the first test we did was the rheological properties. We make sure our product which is the pal oil frond are suitable with the drilling fluid. Some of the lost circulation product or other fluid loss product will give rheological effect to drilling fluids for this case, the oil palm fronds will give increment in rheological properties but the different not so much and still not affect so much to our drilling fluid.

### 4.2 Comparison for 5lb lost circulation materials.

Fan 35 reading (rpm)	Base mud	Base mud + 5lb Nut plug	Base mud + 5lb Palm oil fronds	Base mud + 5lb Corn cob
600	45	49	52	41
300	32	36	39	31
200	26	29	34	26
100	20	23	27	21
6	9	11	13	15
3	8	10	11	15
Plastic viscosity	13	13	13	10
Yield point	19	23	26	21
10sec gel strength	7	10	12	17
10min gel strength	13	18	17	25
Filtration 30minutes/ml	36	27.6	26.4	34
Mud cake thickness, mm	1	2.2	2	2

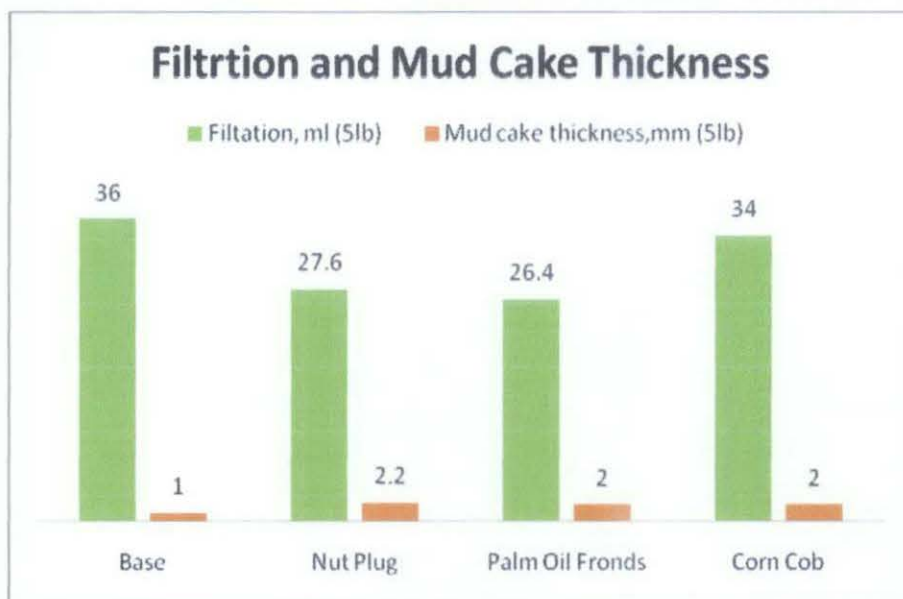
**Table 6: Mud properties of 10.5ppg water base mud with 5lb lost circulation materials**



**Figure 22 :Properties of 10.5 ppg water base mud with different lost circulation materials.**

From the Figure 22, we did comparison between base, nut plug, palm oil fronds and corn cob in plastic viscosity and yield point for the drilling fluid. By adding 5lb of nut plug, palm oil fronds and corn cob to the base mud, we can see the changes of the lost circulation materials to our drilling fluid. Basically when we adding the lost circulation material to our base mud, the yield point of our drilling fluid will increase. Mean that our carrying cutting capacity is better. Compare between nut plug, palm oil fronds and corn cob, palm oil fronds give higher in yield point and plastic viscosities. Follow by nut plug and corn cob. The higher effect in plastic viscosities of palm oil not give so much different between base, nut plug and corn cob. Palm oil frond give 16 cP, nut plug 13 cP and corn cob 10 cP.

Comparison between yield point, after adding the lost circulation material to the base mud the value of yield point will increase. The highest increment is palm oil frond, 27 lb/100ft². In 5lb concentration of lost circulation materials, palm oil frond give better in yield point compare with other lost circulation. Higher in yield point give better carrying capacity to transfer drill cutting to the surface. It shown that palm oil fronds has capabilities to use as lost circulation material in the field.



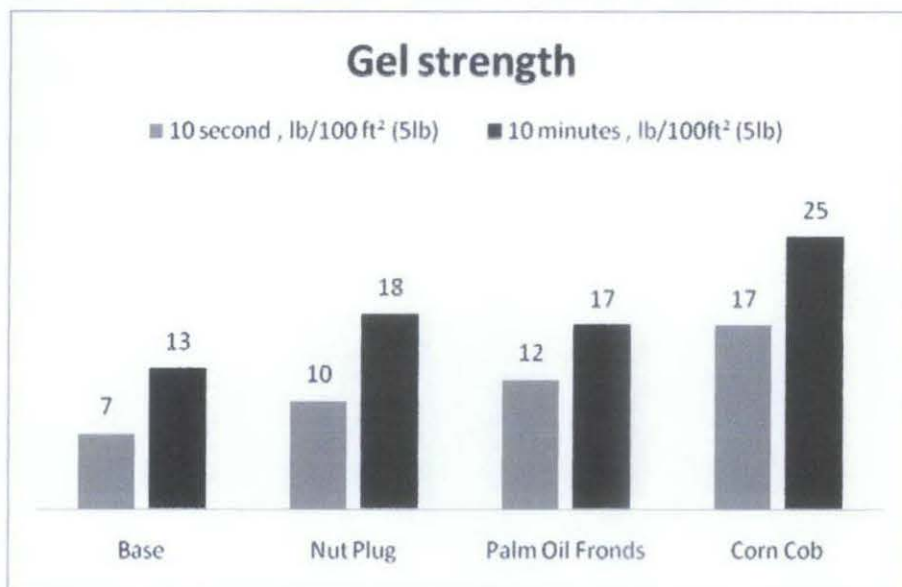
**Figure 23 :Properties of 10.5 ppg water base mud with different lost circulation materials.**

From the Figure 23, It is the comparison in filtration and mud cake thickness. Filtration is most important to our drilling fluid because it can avoid our drilling fluid plug the formation. So much in filtration can cause another problem which is wellbore plugging. Comparing with base, nut plug, palm oil frond and corn cob in the drilling fluid, palm oil fronds give lower fluid loss after 30 minutes, 26.4 ml. For the mud cake thickness, all sample not give so much different in mud cake thickness. All the sample give around 2mm thickness and it is still acceptable in industry. The thickness of mud cake is important because it can effect the differential sticking. In the field, the mud cake should be not so thin and not so thick. It should be on optimum thickness.



**Figure 24: Picture of mudcake after the filtration test.**





**Figure 25 :Properties of 10.5 ppg water base mud with different lost circulation materials.**

Figure 25 discuss the gel strength in our drilling fluid. Gel strength is important to know how much force that we need to rotate again the mud after stop for a while. If we give over force during the rotation, it will cause problem to our drill pipe. There have 2 type of gel strength which are fragile and progressive gel strength. Better gel strength is the fragile gel strengt because the different between 10 seconds and 10 minutes reading is small. Meaning that even we stop the drilling program for a while, the force we need to rotate the bit is lower. Based on the experiment, nut plug and corn cob give so much different in 10 sec and 10 minutes reading. Compare with the palm oil fronds, the different is around 5 value. In this case, palm oil frond give better reading or result in gel strength. The different between 10 seconds and 10 minutes are not so much.

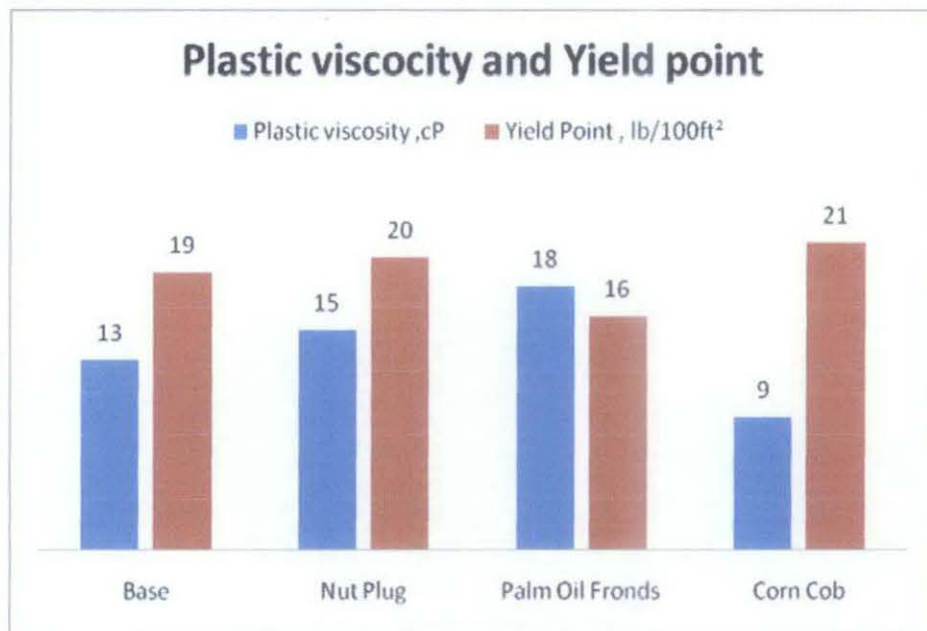


### 4.3 Comparison 10lb/bbl of lost circulation materials

We extend our experiment to evaluate the performance or effect of palm oil frond to the drilling fluid by adding 10lb of lost circulation the based mud. From the test we wan to evaluate the effect of the lost circulation material to our drilling fluid. Theoretically, the properties of drilling fluid will increase when addition of several product. That because we adding more solids to our drilling fluid. So that the properties of the drilling fluid also will change automatically. Most important point we want to see in addition more solids is the effect of plastic viscosity, yield point and filtration of the drilling fluids. How much the effect of the drilling fluid with addition more 5lb solid to the drilling fluids?

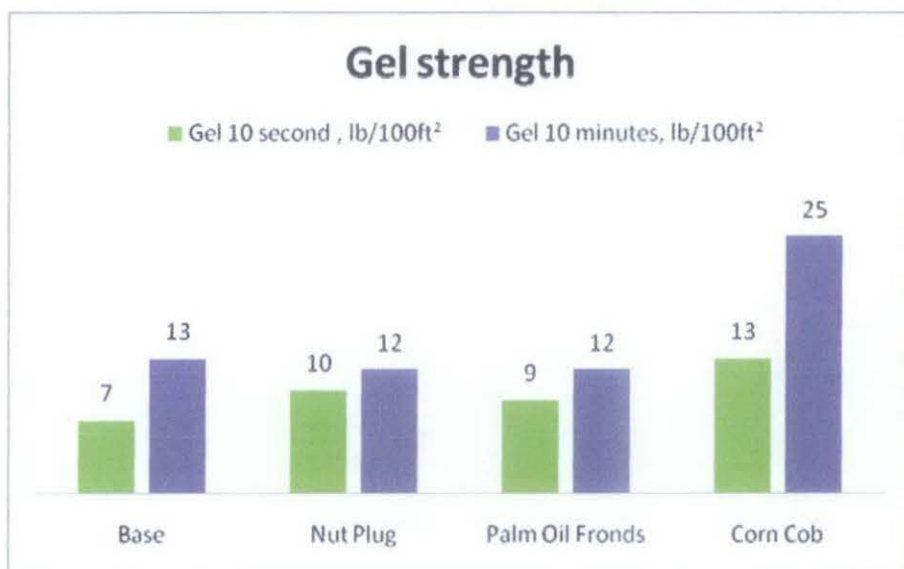
Fan 35 reading (rpm)	Base mud	Base mud + 10lb Nut plug	Base mud + 10lb Palm oil fronds	Base mud + 10lb Corn cob
600	45	50	52	39
300	32	35	34	30
200	26	25	29	25
100	20	20	23	20
6	9	8	10	13
3	8	7	8	12
Plastic viscosity	13	15	18	9
Yield point	19	20	16	21
10sec gel strength	7	10	9	13
10min gel strength	13	12	12	25
Filtration 30minutes/ml	36	30	32	39
Mud cake thickness, mm	1.0	2.0	2.0	2.0

**Table 7: Mud properties of 10.5ppg water base mud with 10lb lost circulation materials.**



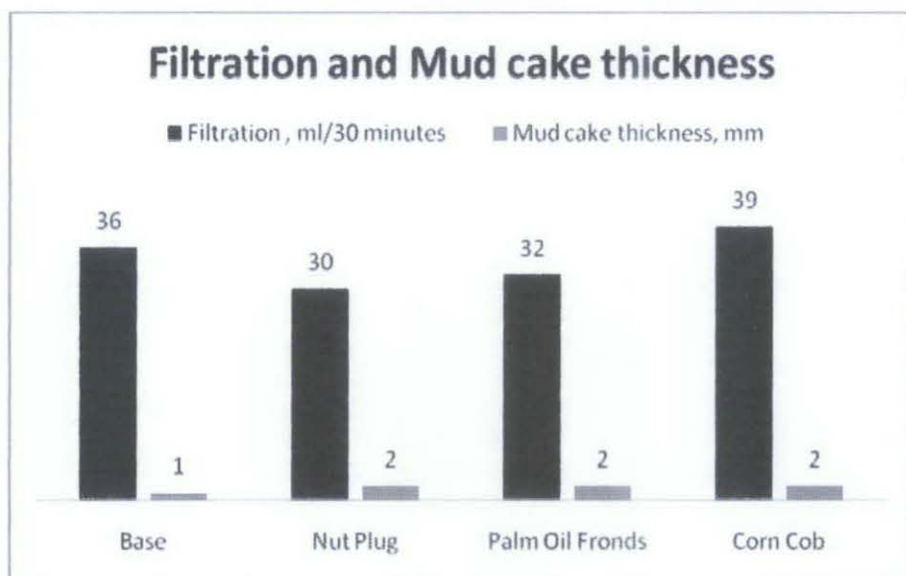
**Figure 26: Mud properties of 10.5ppg water base mud with 10lb lost circulation materials.**

Based on the comparison with 10lb of lost circulation material, palm oil fronds give higher in plastic viscosity compare with nut plug and corn cob. Palm oil frond 18 cP, nut plug 13 cP and corn cob 9 cP. High viscosity in drilling fluid will give some effect during the drilling program. Normally in the field operator will give specification to the services company to design drilling fluid according to their well properties. Observed on yield point for 10lb lost circulation materials effect in drilling fluid, Corn cob give higher effect on yield point. Next goes to nut plug, base mud and lastly oil palm frond. Palm oil frond give lower effect of yield point in 10lb concentration of lost circulation materials. Even though palm oil fronds give lower value of yield point but the difference is small around 4 differences.



**Figure 27: Mud properties of 10.5ppg water base mud with 10lb lost circulation materials.**

Figure 27 is the comparison in gel strength for base, nut plug and palm oil frond and corn cob in drilling fluid. Two type of gel strength are progressive and fragile. Preferable in industry is fragile gel strength. Meaning that, the different between 10 seconds and 10 minutes are small. Based on the experiment result, nut plug is better in gel strength, it gives small changes in 10 sec and 10 min. Palm oil frond also give better in gel strength effect. The difference between the 10 minutes and 10 seconds reading also small around 5 digits. If we plot a graph, there will give small slope value compare with based and corn cob samples. So it will consider under fragile gel strength. An example of progressive gel strength is corn cob because the difference reading quite large.



**Figure 28: Mud properties of 10.5ppg water base mud with 10lb lost circulation materials.**

Figure 23 tell us about the filtration and mud cake thickness for the comparison of 10lb lost circulation material. From the experiment result, Nut plug give lowest filtration quantity after 30 minutes test, 30 ml. The different between the palm oil frond are very small. Palm oil frond also give good effect in filtration test. The worst is corn cob, 39ml. It is more than the filtration of base drilling fluid. In term of mud cake thickness, only base drilling fluid give 1 mm of mud cake thickness. For the nut plug, palm oil frond and corncob the mud cake thickness are 2mm.



4.4 Comparison of concentration and size

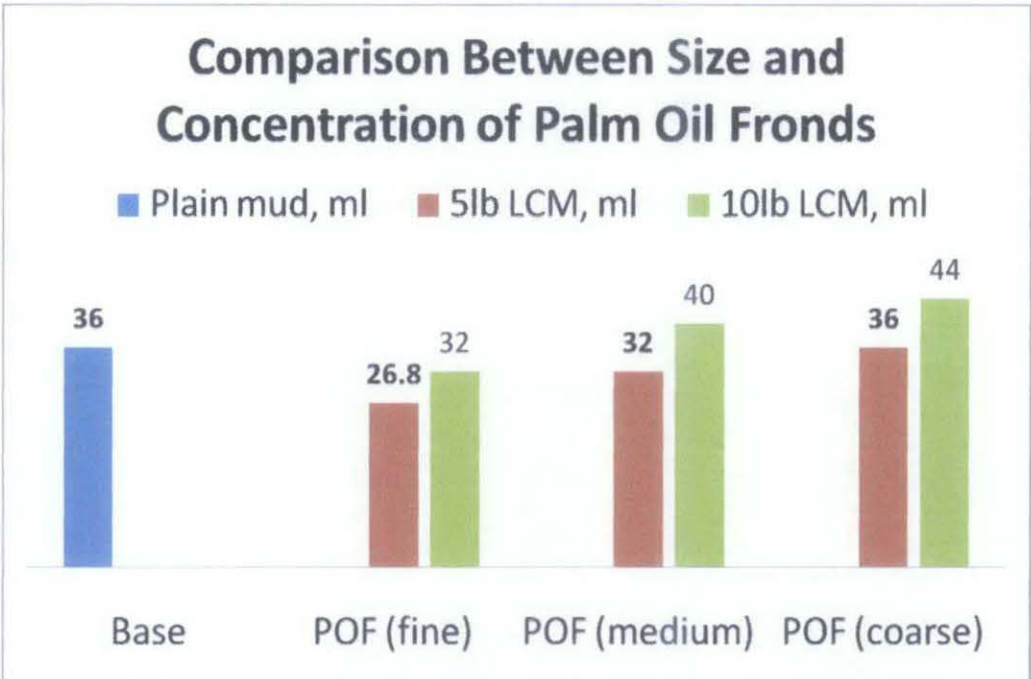


Figure 29: Comparison of palm oil fronds with different concentration and size in drilling fluid.

From the graph above, author need to compare the palm oil fronds effect due to the concentration and size of the sample. The experiment conducted with 0lb, 5lb and 10lb with different size of palm oil fronds. Concentration and size of the lost circulation material also can give affect the performance of palm oil fronds[23].Result from the test, we can consider that the fine of palm oil frond can give better result of filtration in 5lb and 10 lb. In term of the concentration,5lb will give better result in filtration with different size. So from the test we can conclude that the concentration and size also affect the performance of the lost circulation material in drilling fluid. Theoretically, when we adding more lost circulation material in drilling fluid, it will helps to reduce the filtration lost. But in this case, the 5lb of lost circulation give better filtration result compare with the 10lb concentration.

## **CHAPTER 5: CONCLUSION AND RECOMMENDATION.**

For this research we already test the performance of palm oil fronds in the drilling fluid as a lost circulation material. We try to compare their performance with the current lost circulation material which are nut plug and corn cob. The first characteristic of palm oil fronds give better comparison with nut plug and corn cob is in term of price. That because the palm oil fronds is local product and easy to have it and send to the field. Compare with the nut plug, it from other country product and it is quite expensive because of transportation and another services.

From the test, we can conclude that the palm oil fronds still need to continue future studies. Not to say that this product have no characteristic of lost circulation material but before using in oil field industries this product should be in a good characteristic and performance. Based on the comparison, palm oil fronds give approximate result with the nut plug product. The different are slight and still give acceptable result.

For the future studies, author need recommend studies on the palm oil frond in several aspects. Palm oil fronds has potential characteristic for using in oil and gas industries. Author need to recommend the palm oil fronds need to rolling at 170°F. This is to simulate in the drilling fluid in the actual well. So from that test, we can evaluate the rheological properties of our drilling fluid either it change so much or not. Some chemical can effect the rheological properties change extremely when we simulate at the well temperature.

In addition, author need to suggest that, we need focus with the plugging permeability test (PPT) because in the journal said, the PPT test is more accurate because it use screen and it simulate the porosity and permeability of the well bore condition. We can use different micron size to evaluate the effect and different porosity of formation. So from the test we can get the filtration that will enter our formation and hoe much our drilling fluid will affect our formation. From this test, we also can collect the mud cake thickness on the screen.

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## APPENDIXES



## Appendix

No	Details	Week													
		01	02	03	04	05	06	07	08	09	10	11	12	13	14
1	Project work commences														
2	Submission of Progress Report														
3	Project work commences														
4	Pre-EDX combined with seminar														
5	Poster Exhibition														
6	Submission of Final report														
7	Final Oral Presentation														
8	Submission of hardbound copies														

Gantt chart for FYP 2



*Dependable Products From People You Trust*



# **Permeability Plugging Tester - P.P.T.**

**2,000 PSI (13,800 kPa) - 500°F (260°C)**

**Part No. 171-90 (115V)**

**Part No. 171-90-01 (230V)**

## **Instruction Manual**

**Updated 4/23/2010**

**Ver. 2.1**

**OFI Testing Equipment, Inc.**

*11302 Steeplecrest Dr. · Houston, Texas · 77065 · U.S.A.  
Tele: 832.320.7300 · Fax: 713.880.9886 · [www.ofite.com](http://www.ofite.com)*

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## Intro

The Permeability Plugging Tester (PPT) is a modification of the standard 500-mL HTHP Filter press. It may be used in the field or in a laboratory environment. The instrument is useful for performing filtration tests on plugging materials without the interference of particles settling on the filter medium during the heat up process. The PPT is very useful in predicting how a drilling fluid can form a low permeable filter cake to seal off depleted, under pressured intervals and help prevent differential sticking. Typical differential pressures are much higher than those seen in standard HTHP testing.

The pressure cell is similar to those seen in standard HTHP filtration testing, but it is inverted with the filter medium and the back pressure receiver on top of the assembly. The conventional cell may be operated to 2,000 PSI by using hardened steel set screws to secure the cell cap. For elevated pressure, OFITE has designed a special cell with a working pressure of up to 4,000 PSI, pressurized with the conventional hydraulic pump. The cell is pressurized with hydraulic oil and a floating piston separates the oil from the test fluid within the cell.

## Description

The cell is furnished with a standard cell inlet cap, a floating piston, and a scribed outlet cap for the ceramic filter disks. The outlet end of the cell has a 1/4" (6.35 mm) deeper recess than a standard cell to allow for the ceramic disk as a filter medium. The end cap used with the 1/4" (6.35 mm) disk has a scribed concentric pattern rather than the conventional screen. Filter paper or other thin filter medium may be used with this cap by using the spacer ring (Part No. 170-72) to fill the extra 1/4" (6.35 mm) space. Also, an extra thick end cap with a standard screen backup is furnished to use with thin filter media - paper or metal.

All of the end caps are designed to accept the standard valve stem. The inlet, or bottom, valve stem is fitted with a quick-connect for the connection to the hydraulic pressure manifold. The standard hydraulic pressure manifolds are equipped with a 2,000 PSI (13,800 kPa) relief valve. The outlet, or upper, valve stem assembly consists of a dual valve stem with a ball valve in the middle, which facilitates the opening and closing of the outlet flow. Power consumption for the PPT heating jacket is 800 watts.

The 100 mL back pressure receiver is mounted on top of the heating jacket and upside down, when compared to the normal HTHP filter press configuration. It attaches to the cell outlet valve stem and is secured with the retainer pin (Part No. 171-23-1). The fittings on the receiver are reversed with the pressure inlet on the small end (the upper end). A flare fitting may be provided if it is desired to use the low-pressure side of a dual manifold on a nitrogen bottle instead of the standard CO<sub>2</sub> cartridges.

**ecifications**

- Weight: 58 lbs. (26.3 kg)
- Dimensions: 15" x 25" x 42" (38.1 x 63.5 x 106.7 cm)
- Shipping Weight: 84 lbs. (38.1 kg)
- Shipping Dimensions: 30" x 18" x 17" (76.2 x 45.7 x 43.2 cm)
- 800W Heater
- 316 Stainless Steel Test Cell
- Maximum Temperature: 500°F (260°C)
- Maximum Pressure: 2,000 PSI (13,800 kPa)

## omponents

- #171-90 Permeability Plugging Tester; 115V:  
 #171-90-01 Permeability Plugging Tester; 230V:

### Heating Jacket:

- #154-20 Thermometer with Metal Dial; 8" Stem; Dual Scale; 50 - 500°F; 0 - 250°C  
 #164-32 Male Connector for Power Cable (230 Volt Only)  
 #171-00 Heating Jacket; 800W (115 Volt Only)  
 #171-01 Heating Jacket; 800W (230 Volt Only)  
 #171-82 Power Cord with Male Plug; 8'; 16/3 SJ; Round (115 Volt Only)

### Test Cell:

- #170-19 P100 Filter Paper 2½" (6.35 cm)  
 #170-26-1 Hardened Locking Screw; Qty: 12  
 #170-53 2.5" Ceramic Filter Disc; Qty: 10  
 #170-69 Scribed End Cap; 2,500 PSI  
 #170-72 ¼" Spacer for Filter Paper  
 #170-73 Extra Long End Cap  
 #171-02 10" Cell Body; 500 mL  
 #171-21 Cell Cap  
 #171-93 Piston  
 #171-95 T-handle for Piston

### Inlet Pressuring:

- #171-26 #5000 Hose; 3/16" × 3'  
 #171-42 3,000 PSI Gauge; 2½"; ¼" Bottom  
 #171-90-08 Valve Stem; Hydraulics Entry  
 #171-92 2,200 PSI Relief Valve  
 #171-96 Single Speed Handpump  
 #171-96-1 Hydraulic Oil; 1 Quart  
 #171-98 ¼" Ball Valve

### Outlet Pressuring:

- #153-14 Glass Graduated Cylinder; 50 mL × 1 mL  
 #170-04 CO<sub>2</sub> Pressurize Unit  
 #171-10 Back Pressure Receiver; 100 mL (Modified)  
 #171-11 O-ring for Back Pressure Receiver; 100 mL  
 #171-23-1 Safety Pin with Lanyard, 3"  
 #171-90-09 Valve Stem; Filtrate Outlet  
 #171-90-10 Valve Stem; Receiver Entry

### O-rings:

- #170-13-2 Cell O-ring; Qty: 12  
 #170-17 O-ring For Valve Stem; Qty: 12  
 #170-77 O-ring for Stainless Steel Spacer; Qty: 4  
 #171-99 O-ring for Piston; Qty: 4

Valves:

- #170-16 Valve Stem; Qty: 2
- #171-90-08 Valve Stem; Hydraulics Entry
- #171-90-09 Valve Stem; Filtrate Outlet
- #171-90-10 Valve Stem; Receiver Entry; Qty: 2

Tools:

- #170-27 Allen Wrench; 5/32"
- #171-90-15 6" Crescent Adjustable Wrench

**#171-90-SP One Year Spare Parts for #171-90:**

- #143-00-1 Diaphragm for Airco Regulator
- #143-02-13 O-ring for Puncture Pin Holder Assembly; CO<sub>2</sub> Cartridge; Qty: 2
- #143-02-14 O-ring for Puncture Pin Assembly; Qty: 2
- #143-05 \*CO<sub>2</sub> Bulbs EZ Puncture; PKG/10; UN# 1013; Qty: 30
- #143-07 Repair Kit for Regulator
- #153-14 Glass Graduated Cylinder; 50 mL × 1 mL; Qty: 2
- #154-20 Thermometer with Metal Dial; 8" Stem; Dual Scale; 0/500°F; 0/250°C
- #165-44 High-Temperature Thread Lubricant; 1-oz. Tube; Qty: 2
- #170-11 Heating Element; 115V; 200W; Qty: 2
- #170-13-2 Cell O-ring; Buna 90; Qty: 50
- #170-17 O-ring For Valve Stem; Qty: 24
- #170-19 P100 Filter Paper 2½" (6.35 cm); Qty: 5
- #170-26-1 Hardened Locking Screw; Qty: 24
- #170-27 Allen Wrench; 5/32"
- #170-32 1/8" × 1/8" NPT Male Needle Valve
- #170-77 O-ring for Stainless Steel Spacer; Qty: 4
- #171-11 O-ring for Back Pressure Receiver; Qty: 4
- #171-23-1 Safety Pin with Lanyard, 3"; Qty: 2
- #171-90-08 Valve Stem; Hydraulics Entry
- #171-90-09 Valve Stem; Filtrate Outlet
- #171-90-10 Valve Stem; Receiver Entry; Qty: 2
- #171-96-1 Hydraulic Oil; 1 Quart; Qty: 2
- #171-99 O-ring for Piston; Qty: 12

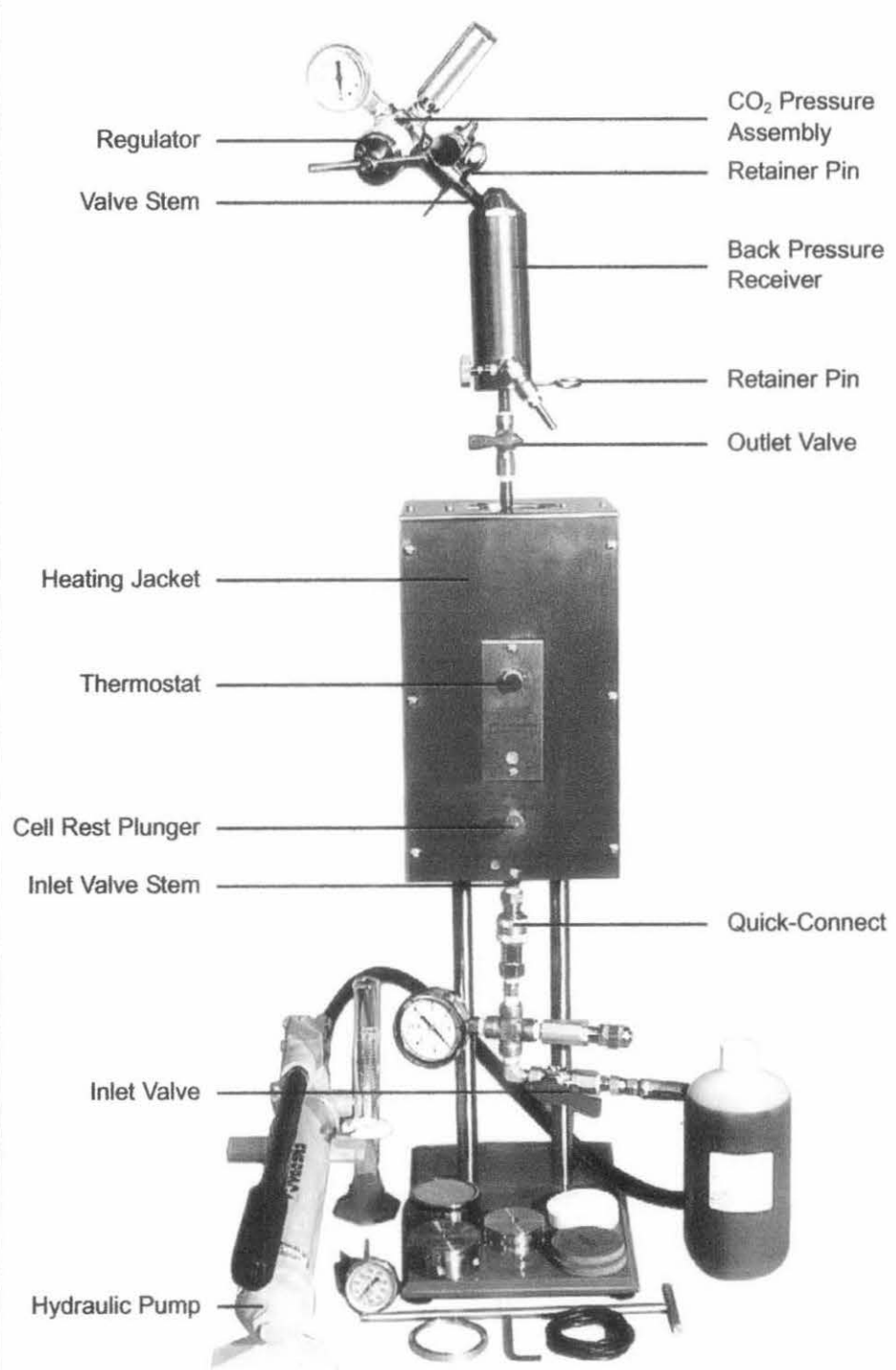
## Safety

1. For safe operation of the Hydraulic Pump Pressurization system, make sure the pressure has been released and the gauge on the pump reads zero before:
  - a. Attempting to disconnect the pressure hose from the cell at the quick-connect.
  - b. Attempting to remove the cell from the heating jacket.
  - c. Reallocating or moving the PPT in the laboratory.
  - d. Refilling the hydraulic pump.
  - e. Performing any maintenance including tightening leaking fittings on the pump, hydraulic fittings, or cell assembly.
2. When refilling or repairing the hydraulic system make sure any spilled oil is cleaned. Oil on the floor is very slippery and can cause falls and injury. Oil spills on the bench can accumulate and become a fire hazard.
3. Always use either nitrogen or carbon dioxide to pressurize the back pressure receiver. Never connect it to compressed air, oxygen, or other non-recommended gas. If nitrogen is used it must be supplied in an approved nitrogen gas cylinder and it must be secured to meet safety standards. CO<sub>2</sub> is normally supplied in small bulbs, which contain about 900 PSI, and are normally used for field operations. Do not allow these bulbs to be heated or exposed to fire as they can explode if overheated.
4. When pressurizing the back pressure receiver always open the supply pressure first, then adjust the regulator. When de-pressurizing, shut off the supply pressure first, then bleed the system of pressure and then back out the regulator T-screw.
5. Cooling the hot cell under water is very dangerous. Be very careful to avoid touching or accidentally dropping the cell. Steam generated when the cell contacts water can cause severe burns.
6. Make sure the electrical source is fused and grounded. Verify the power cord on the heating jacket is in good condition and has the proper ground connection.
7. Electrical problems in the wiring or the heaters may not be obvious by looking at the equipment. A malfunction is indicated if the unit starts blowing fuses or tripping breakers, the heating time seems too long, or the thermostat control does not repeat. These conditions indicate an electrical repair job may be required. Always disconnect the power cables before attempting any repair.



8. The filtration cell assembly is a pressure vessel and these safety precautions should be followed:
  - a. Cell bodies that show signs of stress cracking, severe pitting or have damaged locking screw holes must not be used.
  - b. Cell caps showing evidence of the locking screw holes being pulled or deformed must not be used.

It is strongly recommended the instruction manual be attached to the apparatus and read completely prior to the initial operation by anyone unfamiliar with the equipment.



# Operation

## Preparation



Tip

1. Before starting a test, close all valves and ensure that all regulators are rotated fully counter-clockwise.
2. Connect the heating well to a 110V or 220V AC power source as specified on the nameplate. The heating jacket requires an 800W power supply.
3. Turn the thermostat to about mid-scale to begin heating and place a metal dial thermometer in the thermometer well.

The pilot light will turn on when the heating well is at the temperature setpoint. The temperature should read 10°F (6°C) above the desired test temperature. If it is not, adjust the thermostat.

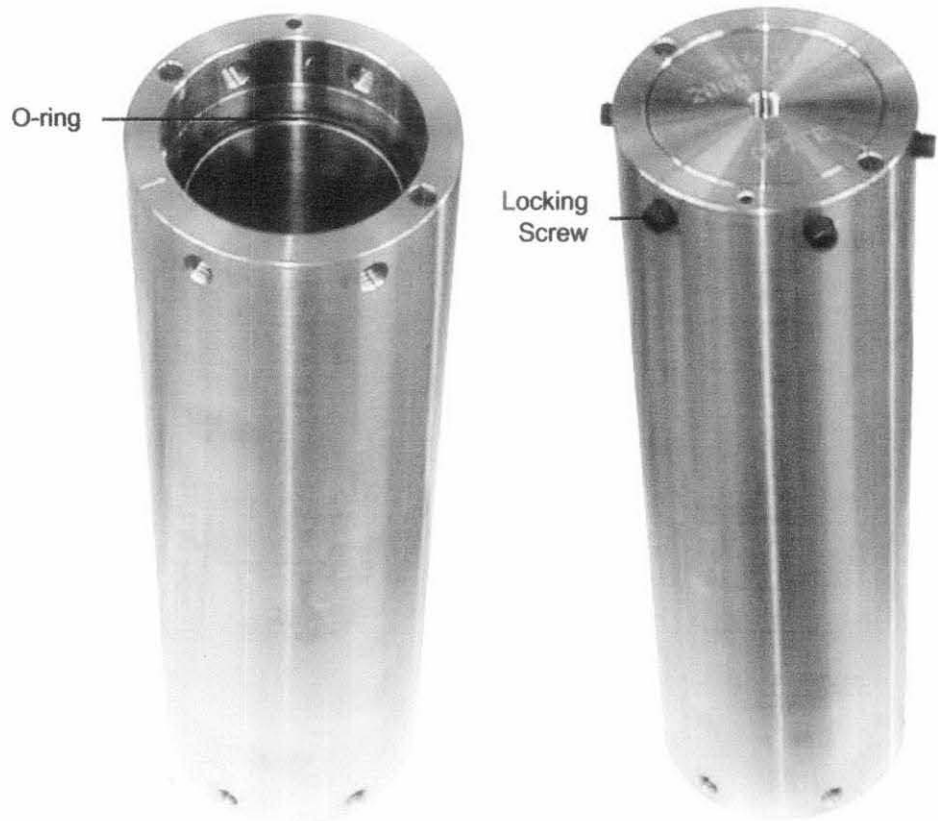
4. Before using the ceramic disk, soak it for at least 10 minutes in base fluid. Use water for freshwater-based fluids, brine for saltwater-based fluids, diesel for oil-based fluids, and a synthetic base for synthetic-based fluids. Never reuse a disk except for return permeability studies. Below is a list of ceramic disks available:

#170-55	Ceramic Filter Disk, 400 milli-darcy, 3 micron, 2½" × ¼"
#170-53-2	Ceramic Filter Disk, 750 milli-darcy, 5 micron, 2½" × ¼"
#170-53-3	Ceramic Filter Disk, 2 darcy, 10 micron, 2½" × ¼"
#170-51	Ceramic Filter Disk, 5 darcy, 20 micron, 2½" × ¼"
#170-53	Ceramic Filter Disk, 10 darcy, 35 micron, 2½" × ¼"
#170-53-1	Ceramic Filter Disk, 20 darcy, 60 micron, 2½" × ¼"
#170-53-4	Ceramic Filter Disk, 100 darcy, 90 micron, 2½" × ¼"

## Operation

### Loading the Filtration Cell

1. Open the cell and check all of the o-rings. Replace any that appear worn or damaged. New o-rings are normally required after each test above 300°F (149°C).
2. Apply a thin coat of silicone grease around the o-rings used on the piston, valve stems, and the cell caps.
3. Position the cell upright with the inlet, or shallow, recess facing upwards. Check the o-ring recess to make sure it is clean. Carefully insert an o-ring (Part No. 170-13) inside the cell recess and cell caps.
4. Find the shorter, screened cell cap and place it into the test cell body. Make sure the arrow on the cell cap lines up with the arrow on the test cell body.
5. Place a locking screw into each of the holes around the top of the test cell body and tighten them with the provided Allen wrench.



6. Push in the red knob located just below the thermostat control on the heating jacket. This moves the stop plunger into position to support the cell while it is being filled with fluid and facilitates installing the outlet cell cap. Invert the cell and place it inside the heating jacket with the inlet cap facing downward.

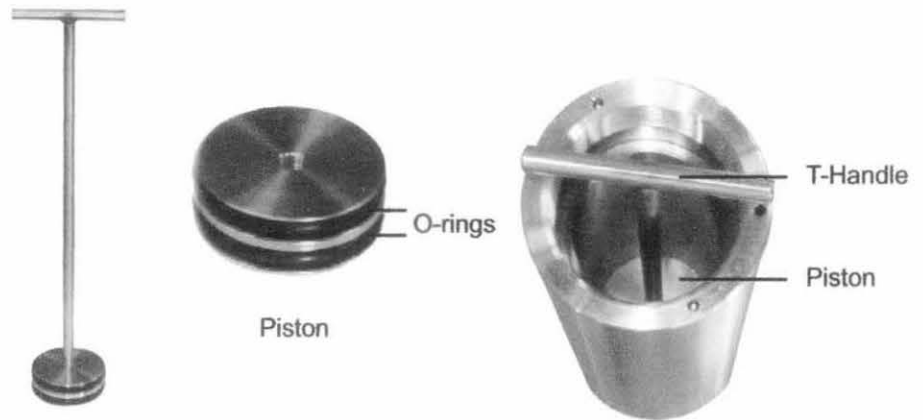
7. Screw the inlet valve stem (with the Quick-Connect fitting) into the inlet cell cap and tighten it completely. Then, open the valve stem by turning it clockwise  $\frac{1}{2}$  to 1 complete turn. When connected, the Quick-Connect end of the valve stem should be pointed down.



8. Connect the hydraulic pump pressurizing hose with the  $\frac{3}{4}$ " (2.0 cm) ball valve and quick-connect fitting to the inlet valve stem assembly.



9. Screw the T-handle into the piston and place it inside the cell, working it up and down to ensure free movement. Position the piston with the T-handle so that it comes into contact with the inlet cell cap.



Piston with T-Handle

10. Open the inlet valve. Turn the pressure release knob on the hydraulic pump clockwise to close the pressure release valve. Stroke the hydraulic pump 6 to 8 times to add approximately  $1\frac{1}{2}$ " (3.81 cm) of hydraulic fluid volume into the cell inlet. Close the inlet valve.



Tip

The best way to determine the volume hydraulic fluid in the cell is to observe the T-handle. When it has risen  $1\frac{1}{2}$ " (3.81 cm), stop adding fluid.

11. Remove the T-handle from the piston and cell.
12. Prepare the test fluid according to API Specification 10.
13. Add approximately 320 mL of test fluid to the cell. Be careful not to pour any fluid on the o-ring recess. The fluid level inside the cell should be flush with the bottom of the o-ring recess.
14. Place the rubber o-ring (Part No. 170-13) in the recess and place the prepared ceramic disk on top of the o-ring.
15. Find the cell cap with the scribed flow lines in the surface. Screw the cell cap into the outlet end of the test cell. Coating the o-ring with a thin coat of high-temperature silicone grease will help.



Tip

*If you are using filter paper instead of a ceramic disk, use the provided screened cell cap to prevent tearing. The screened cap is ¼" thicker to account for the missing disk.*

*If you prefer to use the scribed cell cap with filter paper, use the provided spacer ring to fill in the empty ¼". Place the spacer into the cell on top of the o-ring groove. The o-ring in the spacer should be facing up.*

16. Place a locking screw into each of the holes around the top of the test cell body and tighten them with the provided Allen wrench.



**Outlet Cell Cap  
(Screened) #170-73**



**Outlet Cell Cap  
(Scribed) #170-69**



**Spacer Ring #170-72**

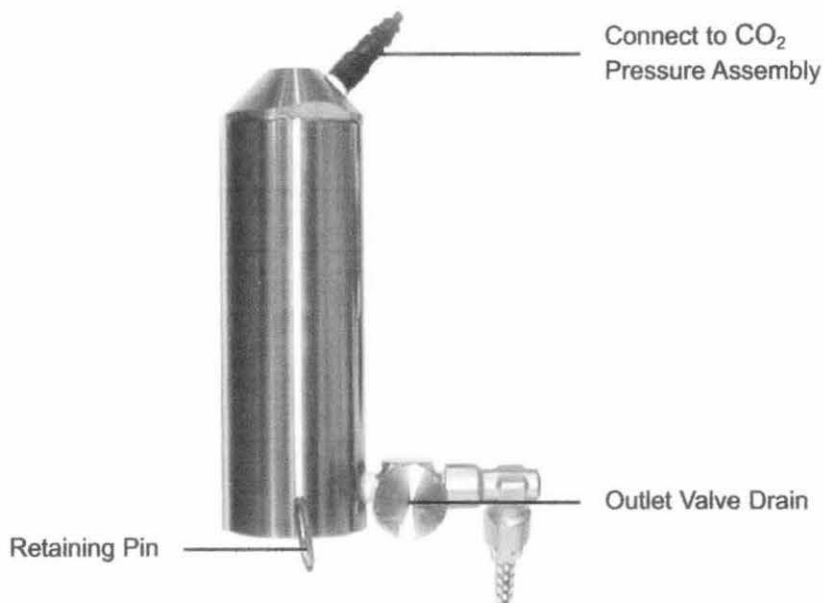


**Inlet Cell Cap  
#171-21**

17. Using a 3-mL syringe, fill the outlet valve with base fluid (water or oil) which will enhance the accuracy of the test. The total dead space volume from the filter media to the back pressure receiver should be filled with the base fluid prior to the test. This will insure that the initial volume of filtrate passing through the filtering media will displace an equal volume of filtrate at the receiver end. In some configurations the dead space can exceed 1 - 2 mL, so erroneous filtrate volumes will result if this dead space is not filled.
18. Install and tighten the outlet valve stem assembly with the 1/8" (0.32 cm) ball valve into the outlet cell cap on top of the cell.
19. Hold the outlet valve assembly with one hand and pull the stop on the heating jacket out of the way to lower the cell fully inside the heating jacket. Rotate the cell until it locks in place over the alignment pin in the bottom of the heating jacket.



20. Close the outlet valve by turning the lever to the 90° position. Place a metal dial thermometer (Part No. 154-20) in the top of the cell in the small hole.
21. Attach the back pressure receiver to the top of the valve assembly. Be careful to not rotate the valve assembly. Lock the receiver in place with the retaining pin. Be sure that the pin is all the way in. The 1/8" (0.32 cm) outlet drain valve on the receiver should be in the closed position.



22. Attach the CO<sub>2</sub> pressure assembly to the valve stem on top of the back pressure receiver and make sure the retainer pin is all the way in.
23. Turn the T-handle on the air regulator counter-clockwise until approximately 6 threads are exposed. Puncture the CO<sub>2</sub> bulb and apply the appropriate amount of back pressure to the receiver for the desired test temperature.
24. While the cell is heating up to the desired temperature, open the inlet valve and apply the amount of pressure indicated in the "Recommended Minimum Back Pressure" table.

Recommended Minimum Back Pressure					
Test Temperature		Vapor Pressure		Minimum Back Pressure	
°F	°C	PSI	kPa	PSI	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
350	177	135	932	160	1,104
400	204	247	1,704	275	1,898
450	232	422	2,912	450	3,105
500	260	680	4,692	700	4,830



Tip



Important

When the closed cell is heating in the jacket, the pressure in the cell will rise rapidly due to the thermal expansion of the sample and the hydraulic fluid. Use the pump to release hydraulic oil and prevent over-pressurization. Maintain the pressure on the fluid until the desired temperature is stabilized, as indicated by the thermometer. Use the hydraulic pump's pressure release valve to regulate and maintain the pressure. The heating time of the sample should never exceed one hour.

**When working with heated pressurized vessels, always wear protective safety glasses.**



Excessive pressure puts stress on four main areas of the cell:

1. End cap bending - may be observed either by eye or by measurement
2. End cap compression - may be observed by the ovalness on the locking screw seats or holes
3. Cylinder shear - elevated areas above the locking screw holes or damaged locking screw holes on the cell body
4. Cylinder stress - stress cracking or severe pitting will appear on the cell body

# Operation

## Test Procedure



1. Once the cell has reached the required temperature, close the valve on the hydraulic pump and open the outlet valve. Operate the pump to increase the pressure in the cell to the desired test pressure to initiate filtration. Using the pump, maintain the desired differential pressure in the cell. The differential pressure is the cell pressure less the amount of back pressure.

**Do not exceed 2,000 PSI as the primary or inlet pressure.**

2. Set a timer for the desired test times. Filtrate should be collected at a minimum of 7.5 and 30 minute intervals. Collect and record the total amount of filtrate and/or mud for 30 minutes. Be sure to maintain the selected differential pressure and test temperature within  $\pm 5^{\circ}\text{F}$  ( $3^{\circ}\text{C}$ ).
3. During filtration collection, the pressure in the cell will tend to decrease, so it will be necessary to apply additional hydraulic pressure to maintain a constant pressure.
4. If the back pressure rises during the test, cautiously reduce the pressure by opening the drain valve on the receiver and drawing off some of the filtrate into the graduated cylinder. The filtrate will be at or near the test temperature and slowly opening the drain valve will minimize any spattering of the fluid and any potential contact with hands and fingers. Bleed only enough to reduce the back pressure to its initial setting.
5. After the 30 minute time period, close the outlet valve. Open the receiver drain valve and allow it to blow dry to remove any filtrate and/or mud from the receiver. Record the total amount of liquid recovered, including the spurt loss.
6. Turn the T-handle on the air regulator clockwise to stop adding pressure to the back pressure receiver.

## Disassembly

1. Release the pressure on the hydraulic pump by turning the release valve on the pump counter-clockwise until the pressure gauge on the hydraulic pump manifold reads 0 PSI (at least four complete turns). Close the inlet valve and remove the hydraulic hose and quick-connect from the inlet valve assembly. Leave the inlet valve assembly connected to the cell.
2. Remove the CO<sub>2</sub> pressuring assembly from the back pressure regulator.
3. Remove the back pressure regulator from the outlet valve assembly.
4. Allow the cell to cool or remove it from the heating jacket and cool it off with cold water.



Important

**The temperature of the sample in the cell must be reduced to less than 100° F (46.5° C) before the cell can be safely opened.**

5. Hold the cell so that the inlet and outlet valves are pointing away from people and equipment. Slowly open the outlet valve stem by turning it one complete turn counter-clockwise. Slowly open the out valve to release the pressure in the cell. Repeat the process with the inlet valve and ensure that all of the pressure has been released from the cell.



Note

The filter cake may block the release of pressure on the outlet side.

6. Turn the cell upside down or lay it on its side. Use either the spanner wrench to remove of the outlet cell cap. Remove the outlet valve assembly and cell cap. Inspect the valve stem to make sure it is not plugged by blowing air into the valve.



Tip

It may be necessary to completely remove any obstructions by inserting a small drill bit, wire, or straightened paper clip, into the valve stem. Make sure the opening is pointed away from people and equipment when inserting the wire.

7. Recover the ceramic disk and very lightly wash the filter cake with the type of base fluid used in the mud (fresh water, brine, diesel, synthetic, etc.) Measure the filter cake to the nearest 1/32" (0.8 mm).

If the ceramic disk does not readily come out of the cell with the flow of fluid:

- a. Lay the cell on its side and over a sink.
- b. Install the hydraulic connector onto the inlet valve stem and open the stem by turning it 2 turns counter-clockwise.



- c. Open the inlet valve and close the pressure relief valve on the hydraulic pump.
- d. Stroke the pump handle 1 to 3 times until the piston pushes the fluid from the cell along with the ceramic disk.

**Do not try to pry or shake the ceramic disk from the cell as it may cause the disk to break.**

- 8. To recover any remaining hydraulic fluid from the cell:
  - a. Screw the T-bar wrench into the piston inside the cell.
  - b. Open the inlet valve and turn the pressure release valve on the hydraulic pump 4 complete turns.
  - c. Manually push the piston to the bottom of the cell.
  - d. Close the pressure release valve on the hydraulic pump and close the inlet valve.
  - e. Remove the hydraulic pump manifold from the inlet valve stem and remove the piston from the cell using the T-handle.
- 9. Completely disassemble the cell and clean and dry the entire apparatus with soap and water. Inspect all o-rings and replace them if necessary.

If you are using brine fluids, clean the outlet valve stem assembly with freshwater and blow it dry with air before re-using.

# Reporting Data

1. The filtrate volume collected should be corrected to a filter area of 7.1 in<sup>2</sup> (4580 mm<sup>2</sup>) so the amount collected will have to be doubled.
2. The spurt loss is defined as the amount of mud and/or filtrate recovered from the collector immediately after the differential pressure is applied until the flow of fluid through the permeable disc stops and gas from the receiver blows out freely. The presence of whole mud in the spurt indicates that there was not an immediate seal of the mud when it passed through the filter. In most cases, the goal is to eliminate or minimize the amount of whole mud in the spurt and in the 30 minute test.
3. Measure the filter cake thickness to the nearest 1/32" (0.8 mm). Although cake descriptions are subjective, such notations such as hard, soft, tough, rubbery, firm, etc. may convey important information of cake quality.

# Calculations

The total PPT fluid loss is calculated as follows:

$$\text{PPT Value (mL)} = \text{Spurt Loss} + (2 \times \text{Fluid Recovered in 30 Minutes})$$

$$\text{Constant Filtration Rate} = \frac{30 \text{ min. filtrate, (mL)} - 7.5 \text{ min. filtrate (mL)}}{2.739}$$

## ***aintenance***

1. Clean the test cell, cell caps, valve stems, and all fittings thoroughly after each test. Make sure all threads are clean and free of debris. Blow air through all valve stems and fittings to clean out any remaining material.
2. Before each test, lubricate all o-rings with high-temperature grease to ensure a proper fit and increased life.
3. Periodically inspect valve stems and locking screws for damage or wear. Replace any that no longer have a sharp point on the end.